# QUALITY ASSURANCE PROJECT PLAN
# Jewett White Lead Site
# Staten Island, New York
# Amendment 1

**Prepared for:**
**United States Environmental Protection Agency/Environmental Response Team**
**Edison, New Jersey**

**By:**
**Lockheed Martin/Scientific Engineering Response & Analytical Services (SERAS)**
**Work Assignment Number: SERAS-138**

**August 2, 2012**

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## QAPP Worksheet #1
## Title and Approval Page

**Site Name/Project Name:** Jewett White Lead Site
**Site Location:** Staten Island, New York

*Document Title:* Quality Assurance Project Plan for Jewett White Lead Site

*Lead Organization:* Environmental Protection Agency/Environmental Response Team (EPA/ERT)

*Preparer's Name and Organizational Affiliation:* Christopher Gussman- Lockheed Martin/Scientific Engineering Response and Analytical Services (SERAS)

*Preparer's Address, Telephone Number, and E-mail Address:* 2890 Woodbridge Avenue, Edison, NJ 08837, (732) 321-4237, christopher.d.gussman@lmco.com

*Preparation Date (Day/Month/Year):* August 2, 2012

Investigative Organization's Project Manager/Date: _____
Signature
Printed Name/Organization: Cheryl Hawkins/ERT Work Assignment Manager

Investigative Organization's Project QA Officer/Date: _____
Signature
Printed Name/Organization: Stephen Blaze/ERT Quality Coordinator

Lead Organization's Project Manager/Date: _____
Signature
Printed Name/Organization: Christopher Gussman/SERAS Task Leader

Approval Signatures/Date: _____
Signature
Printed Name/Title: Deborah A. Killeen/SERAS QA/QC Officer

Approval Authority: Lockheed Martin/SERAS

Other Approval Signatures/Date: _____
Signature
Printed Name/Title: Dennis A. Miller/SERAS Program Manager

Document Control Numbering System: SERAS-138-DQAPPA1-080212

## QAPP Worksheet #2
## QAPP Identifying Information

**Site Name/Project Name:** Jewett White Lead Site
**Site Location:** Staten Island, New York
**Site Number/Code:** A218
**Operable Unit:**
**Contractor Name:** Lockheed Martin
**Contractor Number:** EP-W-09-031

**Contract Title:** SERAS
**Work Assignment Number:** SERAS-138

1. Identify regulatory program: Comprehensive Environmental Response and Compensation Liability Act (CERCLA)

2. Identify approval entity: EPA/ERT

3. The QAPP is (select one): ☐Generic ☒Project Specific

4. List dates of scoping sessions that were held: May 3, 2011, July 17, 2012

5. List dates and titles of QAPP documents written for previous site work, if applicable:

| Title | Approval Date |
|---|---|
| Quality Assurance Project Plan (QAPP) for Jewett White Lead Site, Response Engineering and Analytical Contract (REAC) document # 0388-DQAPP-052909 | 5/29/09 |
| Quality Assurance Project Plan, Jewett White Lead Site, Staten Island, New York, Scientific, Engineering, Response and Analytical Services, SERAS-138-DQAPP-062311 | 06/23/11 |
| | |
| | |

6. List organizational partners (stakeholders) and connection with lead organization:
EPA Region II

7. List data users:
EPA Region II

8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below:

Worksheet #13 – No secondary data are available for these areas.

# QAPP Worksheet #2
# QAPP Identifying Information
# (continued)

| Required QAPP Element(s) and Corresponding QAPP Section(s) | Required Information | Crosswalk to Related Documents |
|---|---|---|
| **Project Management and Objectives** | | |
| 2.1 Title and Approval Page | - Title and Approval Page | 1 |
| 2.2 Document Format and Table of Contents<br>2.2.1 Document Control Format<br>2.2.2 Document Control Numbering System<br>2.2.3 Table of Contents<br>2.2.4 QAPP Identifying Information | - Table of Contents<br>- QAPP Identifying Information | 2 |
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| 2.4 Project Organization<br>2.4.1 Project Organizational Chart<br>2.4.2 Communication Pathways<br>2.4.3 Personnel Responsibilities and Qualifications<br>2.4.4 Special Training Requirements and Certification | - Project Organizational Chart<br>- Communication Pathways<br>- Personnel Responsibilities and Qualifications Table<br>- Special Personnel Training Requirements Table | 5<br>6<br>7<br>8 |
| 2.5 Project Planning/Problem Definition<br>2.5.1 Project Planning (Scoping)<br>2.5.2 Problem Definition, Site History, and Background | - Project Planning Session Documentation (including Data Needs tables)<br>- Project Scoping Session Participants Sheet<br>- Problem Definition, Site History, and Background<br>- Site Maps (historical and present) | 9<br>10 |
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## QAPP Worksheet #2
## QAPP Identifying Information
## (continued)

| Required QAPP Element(s) and Corresponding QAPP Section(s) | Required Information | Crosswalk to Related Documents |
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## QAPP Worksheet #2
## QAPP Identifying Information
## (continued)

| Required QAPP Element(s) and Corresponding QAPP Section(s) | Required Information | Crosswalk to Required Documents |
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## QAPP Worksheet #2
## QAPP Identifying Information
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| Required QAPP Element(s) and Corresponding QAPP Section(s) | Required Information | Crosswalk to Related Documents |
|---|---|---|
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☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #3
## Distribution List

| QAPP Recipients | Title | Organization | Telephone Number | Fax Number | E-mail Address | Document Control Number |
|---|---|---|---|---|---|---|
| Cheryl Hawkins | Work Assignment Manager | ERT | (732) 321-6717 | (732) 321-6724 | Hawkins.CherylA@epamail.epa.gov | SERAS-138-DQAPPA1-080212 |
| Jeff Catanzarita | Acting Work Assignment Manager | ERT | (732) 906-6929 | (732) 321-6724 | Catanzarita.Jeff@epamail.epa.gov | SERAS-138-DQAPPA1-080212 |
| Stephen Blaze | Quality Coordinator | ERT | (732) 906-6921 | (732) 321-6274 | blaze.stephen@epa.gov | SERAS-138-DQAPPA1-080212 |
| Christopher Gussman | Environmental Engineer/Scientist (Phytoremediation) | SERAS | (732) 321-4237 | (732) 494-4021 | christopher.d.gussman@lmco.com | SERAS-138-DQAPPA1-080212 |
| Deborah Killeen | QA/QC Officer | SERAS | (732) 321-4245 | (732) 494-4021 | deborah.a.killeen@lmco.com | SERAS-138-DQAPPA1-080212 |
| Dennis Miller | Program Manager | SERAS | (732) 321-4216 | (732) 494-4021 | dennis.a.miller@lmco.com | SERAS-138-DQAPPA1-080212 |
| Kimberly Staiger | EPA On-Scene Coordinator | EPA R2 | (732) 452-6415 | NA | Staiger.kimberly@epa.gov | SERAS-138-DQAPPA1-080212 |

NA = Not Available

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #4 – June 2011 Event
## Project Personnel Sign-Off Sheet

**Organization:** SERAS/ERT/EPA Region II

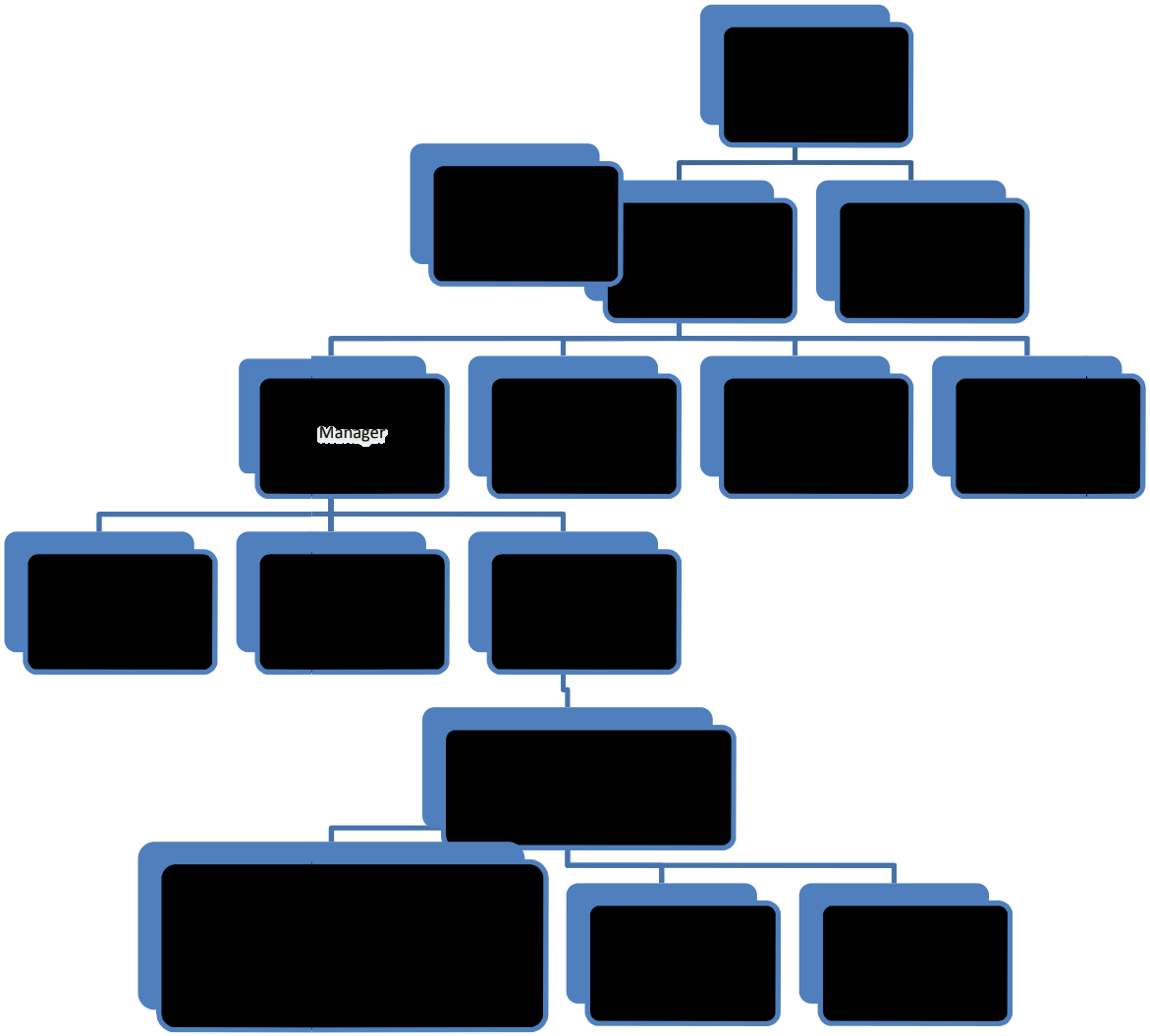
| Project Personnel | Title | Telephone Number | Signature | Date QAPP Read |
|---|---|---|---|---|
| Christopher Gussman | SERAS Environmental Engineer/Scientist (Phytoremediation) | 732-321-4237 | | |
| Kimberly Staiger | EPA On Scene Coordinator | 732-452-6415 | | |
| Jianwei Huang | SERAS Environmental Scientist | 732-321-4233 | | |
| Ebel Martin | SERAS Geophysicist | 732-321-4241 | | |
| Bruce Pullen | SERAS Environmental Technician | 732-321-4282 | | |
| Sandra Richards | SERAS Environmental Technician | 732-321-4265 | | |
| Chris French | SERAS Environmental Technician | 732-494-4040 | | |
| Cheryl Hawkins | EPA/ERT Work Assignment Manager | 732-321-6717 | | |
| Dennis Kalnicky | SERAS AA/XRF Chemist | 732-321-4214 | | |
| Jon McBurney | SERAS Project Engineer | 732-321-4244 | | |
| | | | | |

## QAPP Worksheet #4 – August 2012 Event
## Project Personnel Sign-Off Sheet

**Organization:** SERAS/ERT/EPA Region II

| Project Personnel | Title | Telephone Number | Signature | Date QAPP Read |
|---|---|---|---|---|
| Christopher Gussman | SERAS Environmental Engineer/Scientist (Phytoremediation) | 732-321-4237 | | |
| Kimberly Staiger | EPA On Scene Coordinator | 732-452-6415 | | |
| Bruce Pullen | SERAS Environmental Technician | 732-321-4282 | | |
| Sandra Richards | SERAS Environmental Technician | 732-321-4265 | | |
| Chris French | SERAS Environmental Technician | 732-494-4040 | | |
| Jean Bolduc | SERAS Geologist | 732-321-4280 | | |
| Cheryl Hawkins | EPA/ERT Work Assignment Manager | 732-321-6717 | | |
| Jeff Catanzarita | Technical Liaison | 732-906-6929 | | |

## QAPP Worksheet #5
## Project Organizational Chart



☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #6
## Communication Pathways

| Communication Drivers | Responsible Entity | Name | Phone Number | Procedure (Timing, Pathways, etc.) |
|---|---|---|---|---|
| Approval of initial QAPP and any amendments | ERT Work Assignment Manager<br>ERT Quality Coordinator<br>SERAS Task Leader<br>SERAS Program Manager<br>SERAS QA/QC Officer | Cheryl Hawkins<br>Stephen Blaze<br>Christopher Gussman<br>Dennis Miller<br>Deborah Killeen | (732) 321-6717<br>(732) 906-6921<br>(732) 321-4237<br>(732) 321-4216<br>(732) 321-4245 | SERAS internal peer review, followed by ERT approval, implementation of changes effective only with approved QAPP or QAPP Change Form |
| Nonconformance and Corrective Action | SERAS XRF/AA Chemist<br>SERAS Task Leader<br>ERT Work Assignment Manager<br>SERAS QA/QC Officer | Dennis Kalnicky<br>Christopher Gussman<br>Cheryl Hawkins<br>Deborah Killeen | (732) 321-4214<br>(732) 321-4237<br>(732) 321-6717<br>(732) 321-4245 | Use of the Work Assignment Field Change Form for field issues. Use of laboratory nonconformance memo for XRF screening activities. |
| Posting of Deliverables to ERT-IMS websites | SERAS Task Leader<br>SERAS QA/QC Officer<br>SERAS Deputy Program Manager<br>SERAS Administrative Support | Christopher Gussman<br>Deborah Killeen<br>Richard Leuser<br>Eileen Ciambotti | (732) 321-4237<br>(732) 321-4245<br>(732) 494-4060<br>(732) 321-4255 | As per work assignments, posting of deliverables to ERT-IMS website constitutes delivery to the Work Assignment Manager |
| Work Assignment | SERAS Program Manager | Dennis Miller | (732) 321-4216 | Describes scope of work to SERAS personnel from the ERT Work Assignment Manager |
| Health and Safety On-Site Meeting | SERAS Task Leader and/or Site Health and Safety Officer | Christopher Gussman | (732) 321-4237 | Explains site hazards, personal protective equipment, local hospital |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #7
## Personnel Responsibilities and Qualification Table

| Name | Title | Organizational Affiliation | Responsibilities | Education and Experience Qualifications |
|---|---|---|---|---|
| **July 2011 Sampling Events** | | | | |
| Christopher Gussman | Environmental Engineer/Scientist (Phytoremediation) | SERAS | Project Supervision/Field Investigation, Field Sampling, Field Health and Safety, Reporting | B.S. Biology, M.S. and 14 years plus of environmental experience/Lockheed Martin Employee Files |
| Nesya Belyarchik | Computer Assisted Design (ACAD) | SERAS | Map making | Minimum B.S. degree plus 10 years of related experience/Lockheed Martin Employee Files |
| Mingling Lin | Geographic Information System (GIS) Specialist | SERAS | GIS/Map Making | Minimum B.S. degree plus 3 years of related experience/Lockheed Martin Employee Files |
| Bruce Pullen | Environmental Technician | SERAS | Assist field work, geology support | Experience in environmental sampling/ Lockheed Martin Employee Files |
| Donna Getty | Statistician | SERAS | Assist with statistical support of sampling and data as needed. | Minimum B.S. degree plus 8 years of related experience/Lockheed Martin Employee Files |
| Deborah Killeen | QA/QC Officer | SERAS | QA Oversight | Minimum B.S. degree plus 14 years of related experience/Lockheed Martin Employee Files |
| Martin Ebel | Geologist | SERAS | Assist field work, geological/geophysical support | Minimum B.S. degree plus 14 years of related experience/Lockheed Martin Employee Files |
| Sandra Richards | Environmental Technician | SERAS | Assist field work, sampling, sample processing and management. | Experience in environmental sampling/Lockheed Martin Employee Files |
| Jianwei Huang | Environmental Scientist | SERAS | Assist field work, sampling, sample processing and management. | Minimum PhD. degree plus 8 years of related experience/Lockheed Martin Employee Files |
| Jon McBurney | Project Engineer | SERAS | Water Sampling, field work. Boats | Minimum B.S. degree plus 14 years of related experience/Lockheed Martin Employee Files |
| Christopher French | Environmental Technician | SERAS | Assist field work, sampling, sample processing and management. | Experience in environmental sampling./Lockheed Martin Employee Files |
| Dennis Kalnicky | Chemist | SERAS | XRF of samples | Minimum PhD. degree plus 10 years of related experience/Lockheed Martin Employee Files |
| Richard Leuser | Deputy Program Manager | SERAS | Project Oversight | Minimum B.S. degree plus 8 years of related experience/Lockheed Martin Employee Files |
| Cheryl Hawkins | Work Assignment Manager | EPA/ERT | Technical Direction | Project Management & Coordination Expert/EPA Files |
| Mark Sprenger | Technical Liaison | EPA/ERT | Technical Support | EPA job-related qualifications/EPA Files |

## QAPP Worksheet #7
## Personnel Responsibilities and Qualification Table

| Name | Title | Organizational Affiliation | Responsibilities | Education and Experience Qualifications |
|---|---|---|---|---|
| George Prince | Technical Liaison | EPA/ERT | Technical Support | EPA job-related qualifications/EPA Files |
| Stephen Blaze | Quality Coordinator | EPA/ERT | QA Oversight | EPA job-related qualifications/EPA Files |
| Jeff Catanzarita | Technical Liaison | EPA/ERT | Technical Support | EPA job-related qualifications/EPA Files |
| Jean Bolduc | Geologist | SERAS | Assist in field work | Minimum B.S. degree in Geology plus 8 years of related experience, Professional Geologist |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #8
## Special Personnel Training Requirements Table

| Project Function | Specialized Training – Title or Description of Course | Training Provider | Training Date | Personnel/Groups Receiving Training | Personnel Titles/ Organizational Affiliation | Location of Training Records/Certificates |
|---|---|---|---|---|---|---|
| Sampling | Health & Safety Training | SERAS | November 2010 | Christopher Gussman | Environmental Engineer/Scientist (Phytoremediation)/SERAS | H&S Files |
| Project Oversight | Task Leader Training | REAC | 2002 | Christopher Gussman | Environmental Engineer/Scientist (Phytoremediation)/SERAS | Quality Files |
| QA Oversight | Uniform Federal Policy for Quality Assurance Project Plans | Advanced Systems | January 2006 | Deborah Killeen | QA/QC Officer/SERAS | Quality Files |
| XRF Metals | Demonstration of Capability | SERAS | March 2011 | Dennis Kalnicky | XRF/AA Chemist, SERAS | Quality Files |
| Sampling Activities | 40-Hour Training and/or 8-Hour Refresher Health & Safety Training | SERAS | April 2011<br>October 2010<br>December 2010<br>January 2011<br>October 2010<br>April 2011 | Jianwei Huang<br>Martin Ebel<br>Jon McBurney<br>Bruce Pullen<br>Chris French<br>Sandra Richards | Senior Biologist<br>Geophysicist<br>Process Engineer<br>Environmental Technician<br>Environmental Technician<br>Environmental Technician | H&S Files |
| XRF Metals | Data Integrity Training | SERAS | July 2011 | Dennis Kalnicky | XRF/AA Chemist, SERAS | Quality Files |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #9
## Project Scoping Session Participants Sheet

| | |
|---|---|
| **Project Name: Jewett White Lead Site**<br>**Projected Date(s) of Sampling: July 11through July 31 although scheduling may change based on subcontracting and access.** | **Site Name: Jewett White Lead Site**<br>**Site Location: Staten Island, NY** |

**Date of Session:**
**Scoping Session Purpose: Scoping meeting to discuss field activities**

| Name | Title | Affiliation | Phone # | E-mail Address | Project Role |
|---|---|---|---|---|---|
| Christopher Gussman | Environmental Engineer/Scientist (Phytoremediation)/SERAS | SERAS | 732-321-4237 | christopher.d.gussman@lmco.com | Project Coordination, Oversight of field work. |
| Cheryl Hawkins | WAM | ERT | 732-321-6717 | Hawkins.Cheryla@epa.gov | Work Assignment Manager, Technical Oversight |
| Deborah Killeen | QA/QC Officer | SERAS | 732-321-4245 | deborah.a.killeen@lmco.com | QA Oversight |
| Steve Blaze | EPA/ERT Quality Coordinator | ERT | 732-906-6921 | Blaze.Stephen@epamail.epa.gov | EPA/ERT QA Oversight |
| Jay Patel | ICP, ICP/MS Chemist | SERAS | 732-494-4052 | Jay.r.patel@lmco.com | Inorganic Chemist |
| Larry Martin | Sample Receiving Technician/ Hazardous Waste Coordinator | SERAS | 732-321-4213 | Lawrence.w.martin@lmco.com | Waste Disposal Support |
| Donna Getty | Statistician | SERAS | 732-321-4274 | Donna.j.getty@lmco.com | Statistical Support if needed |
| Richard Leuser | Deputy Program Manager | SERAS | 732-494-4060 | richard.m.leuser@lmco.com | Operations Oversight |

Comments/Decisions: Samples will be collected to satisfy the request by the Region and include Vibracoring of sediments and water offshore of the tugboat property, installation of 3 new flush mount wells on the tugboat property, and collecting Geoprobe cores on the three properties surrounding the tugboat properties. Sediments, soil, and water will be processed by SERAS and submitted to the Region II Laboratory. Soil samples will be analyzed by SERAS personnel using X-ray fluorescence (XRF). 20 percent (%) of XRF samples will be submitted to regional laboratory for confirmation. Samples will be analyzed for TAL metals (no mercury except for sediment 0-6") with an emphasis on Pb. The ERT/WAM will be responsible for Scribe management during sampling. Areas are industrial. The purpose of this study is to further examine the spread and extent of contamination (Pb) from the former Jewett White Lead Site.

It is agreed that the six tugboat locations and 28 (maximum) soil boring locations will just give a general idea as to distribution of lead contamination (e.g. it is not backed by statistical sampling methods). However, this information is still useful to the region to determine extent of contamination.

Consensus Decisions:

Questions which came up during the scoping meeting and answers to these question.

Q: Where did the 28 soil borings number come from?
A: Initially ERT was consulted about developing a sampling plan and 28 was a decided number.

Q: Will we need a geologist to log the cores?
A: Yes

Q: Where in the soil boring from the well installations should we take a sample?
A: From the soil at the water table.

Q: What is the Federal Hazardous Waste #?
A: CERCLIS ID: NYD980531545.

Q: If the property owners want splits, will we be able to take the cores back to Edison for processing?
A: This may be problematic. We will have to see what the property owners request and plan accordingly. For now plan to bring them back to Edison, but be aware that we may need to change that.

Q: For the soil cores, what levels will we be using?
A: We will be using EPA Restricted Use Soil Screening Value, which is 800 ppm.

Q: Can we have a site visit before the field work so we know about the location of obstructions and what we should use to repair the holes?
A: Dependent upon access. This will be arranged once QAPP is finalized. Cold patch will be needed to backfill Geoprobe holes.

Q: Will the tug boats be out of our way when the Vibracore is there?
A: Unknown, dependent upon property owner. We will have to work as best we can with the tug boat facility owners with the Coast Guard's help.

## QAPP Worksheet #9 – August 2012 Event
## Project Scoping Session Participants Sheet

| | |
|---|---|
| **Project Name: Jewett White Lead Site**<br>**Projected Date(s) of Sampling: August** | **Site Name: Jewett White Lead Site**<br>**Site Location: Staten Island, NY** |

**Date of Session: July 17, 2012**
**Scoping Session Purpose: Scoping meeting to discuss field activities**

| Name | Title | Affiliation | Phone # | E-mail Address | Project Role |
|---|---|---|---|---|---|
| Christopher Gussman | Environmental Engineer/Scientist (Phytoremediation)/SERAS | SERAS | 732-321-4237 | christopher.d.gussman@lmco.com | Project Coordination, Oversight of field work. |
| Richard Leuser | Deputy Program Manager | SERAS | 732-494-4060 | richard.m.leuser@lmco.com | Operations Oversight |
| Jeff Catanzarita | WAM | SERAS | 732-906-6929 | catanzarita.jeff@epa.gov | Technical Oversight |
| Donna Getty | Statistician | SERAS | 732-321-4274 | donna.j.getty@lmco.com | QAPP Preparation and Review |
| Eileen Ciambotti | Administrative Support | SERAS | 732-321-4255 | Eileen.t.ciambotti@lmco.com | Administrative support |
| | | | | | |
| | | | | | |
| | | | | | |

Comments/Decisions: One rinsate blank for Pb for the whole event. Surface analyze for all metals, subsurface only interested in lead. Jeff is still waiting to hear whther DESA or CLP will conduct the analyses.

Bathymetric survey – weather dependent; should set aside a week so a day can be chosen with best weather conditions; Chris will talk with sub-contractor who is awarded the bathymetric survey work and get their protocol and attainable resolution.

Consensus Decisions: Need a geologist to log cores

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #10
## Problem Definition

**The problem to be addressed by the project:**

The Jewett White Lead site consists of the historic footprint of the former Jewett White Lead Company facility and the extent of contamination which includes the 1.07-acre parcel of land at 2000-2012 Richmond Terrace and the approximately 4.41-acre parcel of land at 2015 Richmond Terrace (of which, approximately 2.25-acres is not covered by the surface waters of the Kill Van Kull), in the Borough of Staten Island, Richmond County, New York (NY). Historically, John Jewett & Sons White Lead Company operated a white lead manufacturing facility at the Site. John Jewett & Sons White Lead Company owned the Site from 1839 until April 3, 1890 when National Lead & Oil Company of New York (National Lead) acquired the Site property. National Lead continued the manufacture of white lead, until a fire destroyed the plant's main building and storage house in 1920. On December 31, 1943, Moran Towing Corporation acquired the 2015 Richmond Terrace portion of the Site from National Lead. On May 31, 1946, National Lead sold the remaining parcel of land located at 2000 Richmond Terrace. Between 1949 and 1990 various businesses operated at 2000-2012 Richmond Terrace, including Sedutto's Ice Cream factory.

Currently, the property at 2000-2012 Richmond Terrace is fenced and was recently used to store construction equipment and materials from local construction projects. The portion of the Site located at 2015 Richmond Terrace is presently owned by the Moran Towing Corporation, an active tug boat facility.

EPA Region II was contacted in June 2008 to evaluate the Site for possible clean up. The agency collected soil samples to a depth of three feet at the Site in December 2008. Elevated levels of lead (Pb) were found throughout most of the property, both laterally and with depth.. Evidence of surface runoff was apparent along the northern boundary of the Site during the soil sampling event.

EPA/ERT and Lockheed Martin/REAC performed additional sampling and evaluation of surrounding homes and properties, including the railroad trestle, in 2009.

EPA had previously conducted grid-based sampling on the Jewett White Lead Site in October 2010 in support of an Engineering Evaluation/Cost Analysis (EE/CA). Grid sampling will be continued on the neighboring properties to remain consistent with past sampling events.

**July 2011 Sampling Event.** During this event, soil, sediment, groundwater and surface water will be collected. A Direct Push rig will be used to collect up to 28 soil cores, up to a depth of 8-feet, from three properties adjacent to the site. Soil samples will be analyzed for Pb by XRF and 20% (every 5$^{th}$ sample) of the XRF samples will be sent for confirmation by ICP. Vibracore technology will be used to collect six sediment cores to 8-feet below grade within a selected area of the Kill Van Kull adjacent to the site. Sediment collected from the surface 0-6" near each core with a ponar will be retained for possible future toxicological studies. Three additional wells will be installed adjacent to the site and parallel to the Kill Van Kull. Filtered and unfiltered water will be sampled from these three wells and one existing well. Filtered and unfiltered water samples will be collected at four locations within the Kill Van Kull, adjacent to the site and parallel to the bank, at the surface, middle and just above the sediment during a period of low tide. Figure 1 shows approximate sampling locations.

Cores of sediment will be sampled at the surface 0-6", 6-12", and then at one foot intervals to determine depth of contamination. Soil cores will be sampled at one foot intervals.

**The problem to be addressed by the project (continued):**

**August 2012 Sampling Event.** During this event a bathymetric survey of the Moran Towing boat docking area and a portion of the nearby Kill Van Kull will be conducted. The survey will measure depth of water and depth of sediment to bedrock. Results of the bathymetric survey will be used to direct additional sediment sampling in the Kill Van Kull near the Moran Towing facilities docking area. Four to six sediment cores will be collected via vibracores from sediment surface to a depth of 8 feet or to refusal. The boat used for the vibracore collection will have an appropriate anchor system and be large enough to operate safely in the rough waters of the shipping channel. Sediment cores will be processed in the same manner as in the July 2011 sampling event and will be submitted to the EPA Region 2 Laboratory for metals analysis.

The environmental questions being asked:

1) What is the distribution of Pb on selected properties surrounding or on the site?
2) Is Pb contamination migrating off-site and into the surrounding water and sediment?
3) What are the extent, thickness and distribution of sediments in the Kill Van Kull near the Moran Towing facilities docking area?

Observations from any site reconnaissance reports:
None

**A synopsis of secondary data or information from site reports**:
EPA has already determined that a removal action is necessary to address the Pb contamination at the Jewett White Lead site, but needs to continue to delineate the extent of the Pb impacts to determine the most appropriate action (i.e. - capping, excavating, dredging, etc). Previous investigations have determined that Pb extends through the water table at the 2015 Richmond Terrace property with some Pb impacts to the groundwater along the waterfront, but it is not known if the Pb contamination has migrated to neighboring properties or into the surface water and sediments of the Kill Van Kull. In order to select the most appropriate removal action, further investigation is needed.

The possible classes of contaminants and the affected matrices:
**2011Sampling Event:**
Soil– Inductively coupled plasma (ICP) metals, particularly Pb + XRF Pb
Sediment – ICP metals, particularly Pb + Target Analyte List (TAL) metals for the 0-6” interval.
Water- ICP metals, particularly Pb

**August 2012 Sampling Event:**
Sediment – ICP metals, particularly Pb, and Target Analyte List (TAL) metalsl for the 0-6” interval

The rationale for inclusion of chemical and nonchemical analyses:
Monitoring for the purpose of evaluating the distribution of Pb contamination on selected properties surrounding or part of the original site, to see if contamination exists on these properties and how it is distributed. Likewise sampling within the Kill Van Kull will determine if contamination is migrating offsite and into the surrounding water and sediment.

Information concerning various environmental indicators: The goal is to better understand the distribution of contamination within properties surrounding the site and in the sediments in the Kill Van Kull near the Moran Towing docking area.

**Project decision conditions (“If..., then...” statements):** If the concentration of lead exceeds the EPA Region 2 (R2) human health risk assessment number in the soil samples collected from the adjacent properties, then EPA Region 2 will determine what further actions need to be taken in an EE/CA.

If the concentration of lead excceeds the NYSDEC Restricted Use Soil Cleanup Objective (SCO) of 63 mg/kg for the protection of ecological resources, then EPA R2 may conduct future toxicological studies in the sediment samples retained.

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #11
## Project Quality Objectives /Systematic Planning Process Statements

**Who will use the data?** EPA Region 2

What will the data be used for? Data will be used to determine distribution of Pb contamination in soil, sediment, and water samples collected adjacent to the site and in the sediments of the Kill Van Kull near the Moran Towing docking area to determine what action is needed.

**What type of data is needed?** (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)

**July 2011:** ICP metals- soil, sediment and water off-site laboratory. Pb-soil-on-site screening. TAL metals-sediment-offsite laboratory.

**August 2012:**
Sediment – ICP and TAL metals off-site laboratory
Bathymetric survey data measuring depth of water and depth of sediment to bedrock.

**How "good" do the data need to be in order to support the environmental decision?**

Definitive level data for ICP metals are required to meet project objectives. The quantitation limits for metals in various matrices are specified on Worksheet #15. All definitive laboratory analyses will be performed by EPA Region 2 Division of Environmental Science and Assessment (DESA) Laboratory. Worksheets #12 and #28 show the measurement performance criteria that are needed for the quality indicators. Worksheet #20 shows the quality control (QC) samples required. All data analyzed by the EPA R2 DESA laboratory will be validated by EPA Region 2.

Bathymetric data will be collected on-site by the Lockheed Martin (LM) subcontractor Aqua Survey , Inc (ASI) using a survey grade fathometer providing a a vertical resolution of better than 0.1 foot. To ensure system accuracy a bar check will be used to measure depths below the transducer at 5, 10 and 20 feet below water surface at the beginning and end of each survey day.

**How much data are needed? (number of samples for each analytical group, matrix, and concentration)**
**July 2011:**
8: ICP Metals in water from four wells (filtered and unfiltered from each well).
24 : ICP metals in water offshore, 4 locations (parallel to the wells), 3 depths, filtered and unfiltered.
Up to 224: Pb in soil by XRF (ICP metals on 20% for confirmation).
Up to 48: ICP metals in sediment (six locations, from the 0-6", 6-12" intervals and 1-foot increments thereafter up to 8 feet). The surface 0-6" sediment for TAL metals and remaining sediment for ICP metals.

**August 2012:**
Up to 54: ICP metals in sediment (four to six locations, from the 0-6", 6-12" intervals and 1-foot increments thereafter down to 8 feet). The surface 0-6" sediment for TAL metals and remaining sediment for ICP metals.

Where, when, and how should the data be collected/generated?

**July 2011 Sampling Event:** Sediment, water, and soil samples will be collected in 2011 in accordance with the sampling design in Worksheet #17 and the sampling procedures in Worksheet #14. Well installation and sediment collection will occur during the week of 7/11/11. Soil and water sampling will occur either the week of 7/11/11 or the week of 7/18/11. Soil samples will be analyzed for Pb by a SERAS chemist using XRF. Samples containers will be provided to the EPA R2 DESA laboratory. Analytical results for metals will be generated by the EPA Region 2 DESA Laboratory.

**August 2012:**

Bathymetric data will be collected by ASI (Lm subcontractor) in the Kill Van Kull near the Moran Towing facilities docking area using a survey grade fathometer. Bathymetry data will be positioned by RTK-DGPS and interfaced to a personal computer running Hypack 2011 software. Shore-parallel survey lines spaced approximately 20 feet apart will initially be run throughout the study area. Shore-perpendicular lines spaced 200 feet apart will then be surveyed as cross-tie lines

Sub-bottom (depth of sediment) data will be collected by ASI using an ODEC StrataBox system. Sub-bottom data will be collected at 50 foot line spacing with transects oriented perpendicular to the river channel. The positioning system during the sub-bottom survey will be the same as used during the bathymetric survey.

The LM subcontractor, ASI, will conduct vibracore sampling at up to six locations in the Kill Van Kull (see Figure 1). Sediment core samples will be collected from the 0-6", 6-12" intervals and 1-foot increments thereafter down to 8 feet or until refusal. Samples from the 0-6" depth will be analyzed for TAL metals, the remaining depths for ICP metals. Metals analyses will be conducted by the Region 2 DESA Laboratory.

Who will collect and generate the data?

**July 2011.** SERAS subcontractors will advance the sediment and soil cores and SERAS personnel will collect the samples from these cores. SERAS personnel will conduct XRF screening on the soil cores for Pb. Water, sediment and 20% (confirmation) of the soil cores will be relinquished to the EPA Region II DESA laboratory by SERAS personnel.

**August 2012:** SERAS subcontractors will advance the sediment cores and SERAS personnel will collect the samples from these cores. EPA Region 2 DESA Laboratory will generate the analytical results.

Bathymetric and sediment thickness data will be collected by ASI (LM subcontractor).

How will the data be reported?

Hard copy data packages shall contain a table of contents. Data package should be paginated for easy cross reference between the table of contents and relevant portions of the data. Electronic data will be submitted in the Modified Region 2 Electronic Data Deliverables (EDD) format. Data will be sent to SERAS through the WAM.

**August 2012.** Data will be reported in the form of a Final Report which will be prepared in accordance with SERAS SOP#4021, *Preparation of Final Reports.* A draft of the report will be submitted to the WAM for review prior to final delivery.

How will the data be archived?

Data for ICP and/or TAL metals will be archived by the Regional Laboratory in accordance with their standard archival procedures. SERAS will archive results for the XRF screening data in an instrument dedicated logbook. Hard copies of all deliverables will be stored in the SERAS Central Files and e-copies will be stored on SERAS Local Area Network (LAN) and archived in accordance with AP #34, *Archiving Electronic Files*. Data will be imported into a Scribe database and posted to the ERT-Information Management System (IMS) website and archived.

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #12-1-July 2011 and August 2012
## Measurement Performance Criteria Table

| | |
|---|---|
| **Matrix** | Sediment |
| **Analytical Group** | ICP Metals |
| **Concentration Level** | Low |

| Sampling Procedure[1] | Analytical Method/SOP[2] | Data Quality Indicators (DQIs) | Measurement Performance Criteria | QC Sample and/or Activity Used to Assess Measurement Performance | QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A) |
|---|---|---|---|---|---|
| SERAS SOP # 2016 | EPA R2 SOP # C-116 (Prep) EPA SOP# C-109 (ICP-AES) | Accuracy/Bias | <RL | Equipment Blank | S & A |
| | | Accuracy/Bias (lab contamination) | <RL | Method Blank | A |
| | | Accuracy/Bias | $\%R_{AVE} \pm 25\%$ | LCS/LCSD | A |
| | | Precision | RPD ±25% | LCS/LCSD | A |
| | | Accuracy/Bias | %R= 75-125 | MS | A |
| | | Precision | RPD ±35% | Field Duplicate | S & A |

[1]Reference number from QAPP Worksheet #21
[2]Reference number from QAPP Worksheet #23

## QAPP Worksheet #12-2- July 2011
## Measurement Performance Criteria Table

| | |
|---|---|
| **Matrix** | Soil |
| **Analytical Group** | ICP Metals |
| **Concentration Level** | Low |

| Sampling Procedure[1] | Analytical Method/SOP[2] | Data Quality Indicators (DQIs) | Measurement Performance Criteria | QC Sample and/or Activity Used to Assess Measurement Performance | QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A) |
|---|---|---|---|---|---|
| SERAS SOP # 2012 | EPA R2 SOP # C-116 (Prep) EPA SOP# C-109 (ICP-AES) | Accuracy/Bias | <RL | Equipment Blank | S & A |
| | | Accuracy/Bias (lab contamination) | <RL | Method Blank | A |
| | | Accuracy/Bias | $\%R_{AVE} \pm 25\%$ | LCS/LCSD | A |
| | | Precision | RPD ±25% | LCS/LCSD | A |
| | | Accuracy/Bias | %R= 75-125 | MS | A |
| | | Precision | RPD ±35% | Field Duplicate | S & A |

[1]Reference number from QAPP Worksheet #21
[2]Reference number from QAPP Worksheet #23

## QAPP Worksheet #12-3 – July 2011
## Measurement Performance Criteria Table

| | |
|---|---|
| **Matrix** | Water |
| **Analytical Group** | ICP Metals |
| **Concentration Level** | Low |

| Sampling Procedure[1] | Analytical Method/SOP[2] | Data Quality Indicators (DQIs) | Measurement Performance Criteria | QC Sample and/or Activity Used to Assess Measurement Performance | QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A) |
|---|---|---|---|---|---|
| SERAS SOP # 2007 or 2013 | EPA R2 SOP #C-116 (PREP), EPA R2 SOP #C-109 (ICP-AES) or SOP# C-112 (ICP-MS) | Accuracy (lab) | <RL | Method Blank | A |
| | | Precision (laboratory) | ± 20% RPD | LCS/LCSD | A |
| | | Accuracy/Bias (laboratory) | %R= 85-115% | LCS | A |
| | | Accuracy/Bias | %R= 80-120% | MS | A |
| | | Precision | RPD ±20% | Field Duplicate | S&A |

[1]Reference number from QAPP Worksheet #21
[2]Reference number from QAPP Worksheet #23

## QAPP Worksheet #12-4 – July 2011 and August 2012
## Measurement Performance Criteria Table

| | |
|---|---|
| **Matrix** | Sediment |
| **Analytical Group** | Mercury |
| **Concentration Level** | Low |

| Sampling Procedure[1] | Analytical Method/SOP[2] | Data Quality Indicators (DQIs) | Measurement Performance Criteria | QC Sample and/or Activity Used to Assess Measurement Performance | QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A) |
|---|---|---|---|---|---|
| SERAS SOP #2016 | EPA R2 SOP #C-127 | Accuracy/Bias | $\%R_{AVE} \pm 25\%$ | LCS/LCSD | A |
| | | Precision | RPD ± 25% | LCS/LCSD | A |
| | | Accuracy/Bias | %R= 75-125 | MS | A |
| | | Precision | RPD ± 35% | Field Duplicate | S & A |
| | | Accuracy/Bias (Contamination) | <RL | Method Blank | A |

[1]Reference number from QAPP Worksheet #21
[2]Reference number from QAPP Worksheet #23

## QAPP Worksheet #12-5 – July 2011
## Measurement Performance Criteria Table

| | |
|---|---|
| **Matrix** | Soil |
| **Analytical Group** | XRF Metals |
| **Concentration Level** | Low |

| Sampling Procedure[1] | Analytical Method/SOP[2] | Data Quality Indicators (DQIs) | Measurement Performance Criteria | QC Sample and/or Activity Used to Assess Measurement Performance | QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A) |
|---|---|---|---|---|---|
| SERAS SOP# 2012 | SERAS SOP # 1720 | Precision | RSD ± 20% | Laboratory Replicates of D04050 or SRM 2586 | A |
| | | Accuracy/Bias | %R ± 20% of True Value | SRM 2710 + SRM 2711 | A |
| | | Accuracy/Bias | <RL | SRM 2709 | A |

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2)
[2]Reference number from QAPP Worksheet #23 (see Section 3.2)

RPD = Relative Percent Difference
XRF = X-ray fluorescence
SRM = Standard reference Material
Pb = Lead

√ Worksheet Not Applicable

## QAPP Worksheet #13
## Secondary Data Criteria and Limitations Table

| Secondary Data | Data Source (Originating Organization, Report Title, and Date) | Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates) | How Data Will Be Used | Limitations on Data Use |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #14
## Summary of Project Tasks

**July 2011.** Monitoring Well Installation:
Three flush-mount wells will be installed by a SERAS subcontractor to depths ranging from 15-20 feet with a 10-foot screen on the tugboat facility near the site. See Figure #1 for placement. Prior to sampling, the wells will be developed in accordance with SERAS SOP #2044, *Well Development*.

Sampling Tasks:

**July 2011.**
**Groundwater Samples.** Filtered and unfiltered watered samples will be collected from one pre-existing well, denoted as "MSC-1" and three newly installed wells (see Figure 1). Samples will be collected in accordance with SERAS SOP# 2007, *Groundwater Well Sampling* using a portable pump and dedicated tubing. Samples will be submitted for ICP metals analysis to the R2 DESA laboratory.

**Surface Water Samples.** Filtered and unfiltered water samples just off-shore, at multiple depths (surface, middle, and bottom) will be collected at four locations within the Kill Van Kull paralleling the four wells above (see Figure 1) in accordance with SOP # 2013, *Surface Water Sampling* using a Kemmerer bottle or similar direct water sampling method. The samples will be submitted for ICP metals analysis to the R2 DESA laboratory.

**Sediment Samples.** Sediment samples will be collected from the Kill Van Kull using Vibracore technology, at six locations between the two docks at 2015 Richmond Terrace. The six locations will be evenly distributed between the two docks, as roughly indicated in Figure 1. At each of these six locations the core will be collected to 8-feet below grade or refusal. The first 0-6" of sediment will be homogenized. The remaining core will be sampled at 6-12" and in 1-foot increments (up to 9 samples per location), homogenized, sampled for TAL metals and sent to the R2 DESA laboratory. Additional sediment will be collected from the 0-6" interval at approximately the same locations. These samples wil be retained for possible future toxicological studies pending the results of the metals analysis.

**Soil Samples.** Soil samples will be collected from up to 18 locations, along a 100-foot grid, on the surrounding 3 properties, to a depth of 8-feet (Figure 1). The cores will be sampled at every foot for a total of up to 144 samples. These samples will be initially screened using XRF for Pb and 20% (every 5$^{th}$ sample) will be selected and sent to the regional laboratory for confirmation.

Three soil samples will also be collected at the water table during well installation, one soil sample per well, homogenized, and submitted for ICP metals analysis to the R2 DESA laboratory.

**August 2012.** Sediment samples will be collected from the Kill Van Kull using Vibracore technology, at up to six locations extending out from the two docks at 2015 Richmond Terrace. The locations will be chosen based on the bathymetric survey data and sediment depth data. At each of six locations the core will be collected to 8-feet below grade or refusal. The first 0-6" of sediment will be homogenized. The remaining core will be sampled at 6-12" and in 1-foot increments (up to 9 samples per location), homogenized, sampled for TAL metals and sent to the R2 DESA laboratory.

Analysis Tasks:
**July 2011.** Up to 144 soil samples, 52 sediment samples and 32 water samples will be analyzed for metals using the methodology in Worksheet #19.

**August 2012.** Approximately 54 sediment samples will be analyzed for metals using the methodology listed in Worksheet #19.

| |
|---|
| Quality Control Tasks:<br>Appropriate QC samples will be collected and analyzed to meet the requirements in SERAS SOP #2005, *Quality Assurance/Quality Control Samples* and required by the Regional Laboratory. Refer to Worksheets 12 and 28. |
| Secondary Data: NA |
| Data Management Tasks:<br>Field data will be recorded in field notebooks. All analytical data will be retained by the R2 DESA laboratory in accordance with their data management procedures. Data will be sent to the ERT WAM and forwarded to SERAS in an electronic format compatible with Scribe. Scribe will be used for data management. |
| Documentation and Records:<br>All observations noted during field efforts will be documented in accordance with SERAS SOP #4001, *Logbook Documentation* and SERAS SOP #2002, *Sample Documentation*. Documents and records that will be generated during this project include: WP, QAPP, Field Logbooks, Maps, Sample Labels, Field Change Forms, and a Final Report. |
| Assessment/Audit Tasks:<br>No performance audit of field operations is anticipated for this phase of the project. The tasks associated with this QAPP are assessed using management reviews and peer reviews. Management system reviews establish compliance with prevailing management structure, policies and procedures, and ensures that the required data are obtained. Peer review enables the task leader to identify and correct errors before the final deliverable is submitted. |
| Data Review Tasks:<br>All SERAS project deliverables will receive an internal peer review prior to release, per guidelines established in the SERAS AP #22, *Peer Review of SERAS Deliverables*. |

## QAPP Worksheet #15-1 – July 2011
## Reference Limits and Evaluation Table

**Matrix:** Soil
**Analytical Group:** Metals
**Concentration Level:** Low

| Analyte | CAS Number | Project Action Limit [2] | Method CRQLs (mg/kg) | Achievable Laboratory (DESA) Limits[1] MDL (mg/kg) | RL (mg/kg) |
|---|---|---|---|---|---|
| Aluminum | 7429-90-5 | NS | 20 | * | 10 |
| Antimony | 7440-36-0 | NS | 6 | 0.22 | 2 |
| Arsenic | 7440-38-2 | NS | 1 | 0.35 | 0.8 |
| Barium | 7440-39-3 | NS | 20 | 0.24 | 10 |
| Beryllium | 7440-41-7 | NS | 0.5 | 0.02 | 0.3 |
| Cadmium | 7440-43-9 | NS | 0.5 | 0.02 | 0.3 |
| Calcium | 7440-70-2 | NS | 500 | 12.57 | 50 |
| Chromium | 7440-47-3 | NS | 1 | 0.34 | 0.5 |
| Cobalt | 7440-48-4 | NS | 5 | 0.03 | 2 |
| Copper | 7440-50-8 | NS | 2.5 | 0.26 | 1 |
| Iron | 7439-89-6 | NS | 10 | * | 5 |
| Lead | 7439-92-1 | 800 mg/kg | 1 | 0.23 | 0.8 |
| Magnesium | 7439-95-4 | NS | 500 | 5.06 | 50 |
| Manganese | 7439-96-5 | NS | 1.5 | 0.33 | 0.5 |
| Nickel | 7440-02-0 | NS | 4 | 0.09 | 2 |
| Potassium | 7440-09-7 | NS | 500 | 12.36 | 50 |
| Selenium | 7782-49-2 | NS | 3.5 | 0.22 | 2 |
| Silver | 7440-22-4 | NS | 1 | 0.06 | 0.5 |
| Sodium | 7440-23-5 | NS | 500 | 22.48 | 100 |
| Thallium | 7440-28-0 | NS | 2.5 | 3.14 | 2 |
| Vanadium | 7440-62-2 | NS | 5 | 0.40 | 2 |
| Zinc | 7440-66-6 | NS | 6 | 1.57 | 2 |

[1] MDL study cannot be successfully performed on these analytes because of high background levels in matrix (sand).
[2] EPA Region 2 Human Health Risk Number
NS = Not Specified
mg/kg = milligrams per kilogram

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #15-2 – July 2011 and August 2012
## Reference Limits and Evaluation Table

**Matrix:** Sediment
**Analytical Group:** Metals
**Concentration Level:** Low

| Analyte | CAS Number | Project Action Limit [2] | Method CRQLs (mg/kg) | Achievable Laboratory (DESA) Limits[1] | |
|---|---|---|---|---|---|
| | | | | MDL (mg/kg) | RL (mg/kg) |
| Aluminum | 7429-90-5 | NS | 20 | * | 10 |
| Antimony | 7440-36-0 | NS | 6 | 0.22 | 2 |
| Arsenic | 7440-38-2 | NS | 1 | 0.35 | 0.8 |
| Barium | 7440-39-3 | NS | 20 | 0.24 | 10 |
| Beryllium | 7440-41-7 | NS | 0.5 | 0.02 | 0.3 |
| Cadmium | 7440-43-9 | NS | 0.5 | 0.02 | 0.3 |
| Calcium | 7440-70-2 | NS | 500 | 12.57 | 50 |
| Chromium | 7440-47-3 | NS | 1 | 0.34 | 0.5 |
| Cobalt | 7440-48-4 | NS | 5 | 0.03 | 2 |
| Copper | 7440-50-8 | NS | 2.5 | 0.26 | 1 |
| Iron | 7439-89-6 | NS | 10 | * | 5 |
| Lead | 7439-92-1 | 31 mg/kg | 1 | 0.23 | 0.8 |
| Magnesium | 7439-95-4 | NS | 500 | 5.06 | 50 |
| Manganese | 7439-96-5 | NS | 1.5 | 0.33 | 0.5 |
| Mercury | 7439-97-6 | NS | 0.1 | .0043 | 0.05 |
| Nickel | 7440-02-0 | NS | 4 | 0.09 | 2 |
| Potassium | 7440-09-7 | NS | 500 | 12.36 | 50 |
| Selenium | 7782-49-2 | NS | 3.5 | 0.22 | 2 |
| Silver | 7440-22-4 | NS | 1 | 0.06 | 0.5 |
| Sodium | 7440-23-5 | NS | 500 | 22.48 | 100 |
| Thallium | 7440-28-0 | NS | 2.5 | 3.14 | 2 |
| Vanadium | 7440-62-2 | NS | 5 | 0.40 | 2 |
| Zinc | 7440-66-6 | NS | 6 | 1.57 | 2 |

[1] MDL study cannot be successfully performed on these analytes because of high background levels in matrix (sand).
[2] EPA Region 2 Human Health Risk Number
NS = Not Specified
mg/kg = milligrams per kilogram

## QAPP Worksheet #15-3 – July 2011
## Reference Limits and Evaluation Table

**Matrix:** Aqueous
**Analytical Group:** Metals-ICP
**Concentration Level:** Low

| Analyte | CAS Number | Project Action Limit[2] | Method CRQLs (µg/L) | Achievable Laboratory (DESA) Limits[1] | |
|---|---|---|---|---|---|
| | | | | MDLs (µg/L) | RLs (µg/L) |
| Aluminum | 7429-90-5 | NS | 200 | 41.02 | 100 |
| Antimony | 7440-36-0 | NS | 60 | 2.287 | 20 |
| Arsenic | 7440-38-2 | NS | 10 | .881 | 8 |
| Barium | 7440-39-3 | NS | 200 | 4.094 | 6 |
| Beryllium | 7440-41-7 | NS | 5 | 0.543 | 3 |
| Cadmium | 7440-43-9 | NS | 5 | 0..326 | 3 |
| Calcium | 7440-70-2 | NS | 5000 | 21.64 | 500 |
| Chromium | 7440-47-3 | NS | 10 | .253 | 5 |
| Cobalt | 7440-48-4 | NS | 50 | 1.257 | 20 |
| Copper | 7440-50-8 | NS | 25 | 2.304 | 10 |
| Iron | 7439-89-6 | NS | 100 | 3.641 | 50 |
| Lead | 7439-92-1 | 50 | 10 | .515 | 8 |
| Magnesium | 7439-95-4 | NS | 5000 | 154.2 | 500 |
| Manganese | 7439-96-5 | NS | 15 | .986 | 5 |
| Mercury | 7439-97-6 | NS | 0.2 | NS | NS |
| Nickel | 7440-02-0 | NS | 40 | 1.374 | 20 |
| Potassium | 7440-09-7 | NS | 5000 | 112.3 | 500 |
| Selenium | 7782-49-2 | NS | 35 | 2.448 | 20 |
| Silver | 7440-22-4 | NS | 10 | 1.05 | 5 |
| Sodium | 7440-23-5 | NS | 5000 | 54.66 | 1000 |
| Thallium | 7440-28-0 | NS | 25 | 2.168 | 20 |
| Vanadium | 7440-62-2 | NS | 50 | 2.466 | 20 |
| Zinc | 7440-66-6 | NS | 60 | 2.185 | 20 |

[1]Based on MDLs and RLs from EPA R2 DESA Laboratory
[2]Value based on NYSDEC Part 703: Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations, Table 3. Maximum Allowable Concentration
NS – Not Specified

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #16
## Project Schedule Timeline Table

| Activities | Organization | Dates (MM/DD/YY) | | Deliverable | Deliverable Due Date |
|---|---|---|---|---|---|
| | | Anticipated Date(s) of Initiation | Anticipated Date of Completion | | |
| Field Work- Well installation, water sampling, vibracoring | SERAS | 07/11/11 | 07/31/11 | Technical Memorandum | 28 days after all field work is completed and data received |
| Field Work- Soil Sampling, Well Sampling | SERAS | 07/11/11 | 07/31/11 | Technical Memorandum | 28 days after all field work is completed and data received |
| Field Work – Bathymetric Survey | ASI | August 2012 | August 2012 | Contour Maps | TBD |
| Field Work – Vibracore Sampling | ASI | August 2012 | August 2012 | NA | NA |
| Metals Analysis | Region 2 DESA | August 2012 | TBD | Preliminary Analytical Results | 30 business days after receipt of last sample |
| Validation of Metals Results | Region 2 DESA | TBD | TBD | Validated EDD | 30 business days after receipt of last data package |
| Draft Final Report | SERAS | TBD | 15 working days after receipt of validated laboratory results | TBD | 15 working days after receipt of validated laboratory results |
| Final Report | SERAS | TBD | 5 days following reciept of comments from the WAM | TBD | 5 days following reciept of comments from the WAM |

Note that there is a large effort coordinating EPA, subcontractors, and property owners. Access for this field effort may easily effect dates indicated.

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #17
## Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):
**July 2011.** EPA has already determined that a removal action is necessary to address the lead contamination at the Jewett White Lead site, but there is a need to continue to delineate the extent of the lead in support of an Engineering Evaluation/Cost Analysis (EE/CA) to determine the most appropriate removal action (i.e. – capping, excavating, dredging, etc). It is currently known that lead extends through the water table at the 2015 Richmond Terrace property with some lead impacts to the groundwater along the waterfront, but it is not known whether the lead contamination has migrated to neighboring properties or into the surface water and sediments of the Kill Van Kull. Further sampling is being performed to determine if elevated levels of metals (Pb) are located on these sites and at what locations.

A grid system (100-foot centers) was selected for the soil sample locations. Six sediment locations, four well samples, and four off shore water samples adjacent to these wells will be collected to determine if Pb is entering the Kill Van Kull.

The six sediment sample locations were selected to be evenly distributed within the sediment in the Kill Van Kull adjacent to the property. It was assumed that these six sediment locations would well represent the sediment adjacent to the property.

Groundwater samples will be collected at four locations parallel to the Kill Van Kull. Four evenly spaced locations were chosen to better intercept potential groundwater plumes flowing across the property from the site towards the Kill Van Kull.

Four locations within the Kill Van Kull, times 3 depths per location, parallel to the four wells, have been selected to determine if there is Pb contamination in the water of the Kill Van Kull and to better determine if this contamination is resulting from contaminated groundwater entering the Kill Van Kull from the Site or the result of another source.

Grid sampling will be used to delineate the extent of contamination in soil and define concentration gradients on the neighboring properties.

**August 2012.** Up to six vibracore sampling locations will be chosen from within the Kill Van Kull based on the results of the bathymetric survey and depth of sediment surveys..

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [May refer to map or Worksheet #18 for details]:

**July 2011**. Soil will be sampled, at several depths, to determine the presence and distribution of Pb contamination from the site within soils at the selected properties.

Groundwater will be collected at four wells to determine if Pb contamination is migrating in groundwater from the site towards the Kill Van Kull.

Water samples will be collected within the Kill Van Kull to determine if Pb contamination may be found within the water of the Kill Van Kull. Sampling at multiple depths will help to determine, if there is Pb contamination in the water, from where it is originating. Sediment samples will be collected within the Kill Van Kull to determine if there is Pb contamination within the sediments and to what depth.

**August 2012**. Similarly to the July 2011 event, sediment samples will be collected from the Kill Van Kull to determine if there is Pb contamination within the sediments and to what depth. For this sampling event, cores will be taken extending out into the Kill Van Kull in the vicinity of the two docks at the Moran Towing facilities.

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| **May-June 2011 Sampling Events** | | | | | | | |
| JWL-SED1-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED1-B | Sediment | 6-12 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED1-C | Sediment | 12-24 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED1-D | Sediment | 24-36 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED1-E | Sediment | 36-48 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED1-F | Sediment | 48-60 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED1-G | Sediment | 60-72 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED1-H | Sediment | 72-84 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED1-I | Sediment | 84-96 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-SED2-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED2-B | Sediment | 6-12 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED2-C | Sediment | 12-24 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED2-D | Sediment | 24-36 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED2-E | Sediment | 36-48 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED2-F | Sediment | 48-60 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED2-G | Sediment | 60-72 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED2-H | Sediment | 72-84 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED2-I | Sediment | 84-96 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED3-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED3-B | Sediment | 6-12 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-SED3-C | Sediment | 12-24 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED3-D | Sediment | 24-36 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED3-E | Sediment | 36-48 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED3-F | Sediment | 48-60 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED3-G | Sediment | 60-72 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED3-H | Sediment | 72-84 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED3-I | Sediment | 84-96 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED4-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED4-B | Sediment | 6-12 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED4-C | Sediment | 12-24 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED4-D | Sediment | 24-36 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-SED4-E | Sediment | 36-48 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED4-F | Sediment | 48-60 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED4-G | Sediment | 60-72 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED4-H | Sediment | 72-84 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED4-I | Sediment | 84-96 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED5-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED5-B | Sediment | 6-12 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED5-C | Sediment | 12-24 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED5-D | Sediment | 24-36 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED5-E | Sediment | 36-48 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED5-F | Sediment | 48-60 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-SED5-G | Sediment | 60-72 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED5-H | Sediment | 72-84 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED5-I | Sediment | 84-96 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED6-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED6-B | Sediment | 6-12 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED6-C | Sediment | 12-24 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED6-D | Sediment | 24-36 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED6-E | Sediment | 36-48 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED6-F | Sediment | 48-60 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED6-G | Sediment | 60-72 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-SED6-H | Sediment | 72-84 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-SED6-I | Sediment | 84-96 | ICP Metals | Low | 1 | SERAS SOP # 2016 | Judgmental (6 Locations evenly spaced) |
| JWL-Well2Soil | Soil | At Water Table | TAL Metals | Low | 1 | SERAS SOP # 2012 | Sampling at water table during well installation. |
| JWL-Well3Soil | Soil | At Water Table | TAL Metals | Low | 1 | SERAS SOP # 2012 | Sampling at water table during well installation. |
| JWL-Well4Soil | Soil | At Water Table | TAL Metals | Low | 1 | SERAS SOP # 2012 | Sampling at water table during well installation. |
| JWL-Well1-Filtered | Water | N/A | ICP Metals | Low | 1 | SERAS SOP # 2007 | Judgmental |
| JWL-Well1-Unfiltered | Water | N/A | ICP Metals | Low | 1 | SERAS SOP # 2007 | Judgmental |
| JWL-Well2-Filtered | Water | N/A | ICP Metals | Low | 1 | SERAS SOP # 2007 | Judgmental |
| JWL-Well2-Unfiltered | Water | N/A | ICP Metals | Low | 1 | SERAS SOP # 2007 | Judgmental |
| JWL-Well3-Filtered | Water | N/A | ICP Metals | Low | 1 | SERAS SOP # 2007 | Judgmental |
| JWL-Well3-Unfiltered | Water | N/A | ICP Metals | Low | 1 | SERAS SOP # 2007 | Judgmental |
| JWL-Well4-Filtered | Water | N/A | ICP Metals | Low | 1 | SERAS SOP # 2007 | Judgmental |
| JWL-Well4-Unfiltered | Water | N/A | ICP Metals | Low | 1 | SERAS SOP # 2007 | Judgmental |
| JWL-KVK1-TOP-F | Water | Surface (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-KVK1-TOP-UF | Water | Surface (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK1-MID-F | Water | Midlevel (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK1-MID-UF | Water | Midlevel (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK1-BOT-F | Water | Bottom, near sediment (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK1-BOT-UF | Water | Bottom, near sediment (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK2-TOP-F | Water | Surface (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK2-TOP-UF | Water | Surface (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK2-MID-F | Water | Midlevel (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK2-MID-UF | Water | Midlevel (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK2-BOT-F | Water | Bottom, near sediment (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK2-BOT-UF | Water | Bottom, near sediment (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-KVK3-TOP-F | Water | Surface (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK3-TOP-UF | Water | Surface (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK3-MID-F | Water | Midlevel (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK3-MID-UF | Water | Midlevel (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK3-BOT-F | Water | Bottom, near sediment (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK3-BOT-UF | Water | Bottom, near sediment (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK4-TOP-F | Water | Surface (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK4-TOP-UF | Water | Surface (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK4-MID-F | Water | Midlevel (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK4-MID-UF | Water | Midlevel (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-KVK4-BOT-F | Water | Bottom, near sediment (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-KVK4-BOT-UF | Water | Bottom, near sediment (at low tide) | ICP Metals | Low | 1 | SERAS SOP # 2013 | Judgmental (in Kill Van Kull parallel to the sampled wells) |
| JWL-Soil1-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil1-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil1-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil1-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil1-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil1-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil1-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil1-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil2-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil2-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil2-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil2-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil2-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil2-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil2-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil2-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil3-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil3-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil3-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil3-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil3-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil3-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil3-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil3-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soi4-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil4-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil4-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil4-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil4-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil4-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil4-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil4-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil5-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil5-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil5-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil5-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil5-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil5-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil5-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil5-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil6-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil6-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil6-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil6-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil6-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil6-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil6-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil6-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil7-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil7-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil7-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil7-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil7-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil7-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil7-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil7-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil8-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil8-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil8-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil8-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil8-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil8-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil8-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil8-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil9-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil9-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil9-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil9-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil9-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil9-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil9-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil9-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil10-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil10-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil10-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil10-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil10-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil10-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil10-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil10-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil11-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil11-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil11-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil11-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil11-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil11-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil11-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil11-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil12-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil12-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil12-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil12-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil12-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil12-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil12-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil12-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil13-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil13-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil13-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil13-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil13-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil13-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil13-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil13-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil14-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil14-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil14-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil14-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil14-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil14-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil14-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil14-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil15-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil15-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil15-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil15-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil15-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil15-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil15-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil16-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil16-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil16-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil16-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil16-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil16-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil16-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil16-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil17-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil17-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil17-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil17-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil17-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil17-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil17-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil17-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil18-A | Soil | 0-12 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil18-B | Soil | 12-24 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil18-C | Soil | 24-36 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil18-D | Soil | 36-48 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil18-E | Soil | 48-60 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil18-F | Soil | 60-72 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| JWL-Soil18-G | Soil | 72-84 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-Soil18-H | Soil | 84-96 | Pb (20% for ICP Metals confirmation) | Low | 1 | SERAS SOP # 2012 | Systematic grid |
| **July –August 2012** | | | | | | | |
| JWL-SED7-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP #2016 | Biased – Based on bathymetric survey results |
| JWL-SED7-B<br>JWL-SED7-C<br>JWL-SED7-D<br>JWL-SED7-E<br>JWL-SED7-F<br>JWL-SED7-G<br>JWL-SED7-H<br>JWL-SED7-I | Sediment | 6-12<br>12-24<br>24-36<br>36-48<br>48-60<br>60-72<br>72-84<br>84-96 | ICP Metals | Low | 1 per depth or until refusal | SERAS SOP #2016 | Biased – Based on bathymetric survey results |
| JWL-SED8-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP #2016 | Biased – Based on bathymetric survey results |
| JWL-SED8-B<br>JWL-SED8-C<br>JWL-SED8-D<br>JWL-SED8-E<br>JWL-SED8-F<br>JWL-SED8-G<br>JWL-SED8-H<br>JWL-SED8-I | Sediment | 6-12<br>12-24<br>24-36<br>36-48<br>48-60<br>60-72<br>72-84<br>84-96 | ICP Metals | Low | 1 per depth or until refusal | SERAS SOP #2016 | Biased – Based on bathymetric survey results |
| JWL-SED9-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP #2016 | Biased – Based on bathymetric survey results |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-SED9-B<br>JWL-SED9-C<br>JWL-SED9-D<br>JWL-SED9-E<br>JWL-SED9-F<br>JWL-SED9-G<br>JWL-SED9-H<br>JWL-SED9-I | Sediment | 6-12<br>12-24<br>24-36<br>36-48<br>48-60<br>60-72<br>72-84<br>84-96 | ICP Metals | Low | 1 per depth or until refusal | SERAS SOP #2016 | Biased – Based on bathymetric survey results |
| JWL-SED10-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP #2016 | Biased – Based on bathymetric survey results |
| JWL-SED10-B<br>JWL-SED10-C<br>JWL-SED10-D<br>JWL-SED10-E<br>JWL-SED10-F<br>JWL-SED10-G<br>JWL-SED10-H<br>JWL-SED10-I | Sediment | 6-12<br>12-24<br>24-36<br>36-48<br>48-60<br>60-72<br>72-84<br>84-96 | ICP Metals | Low | 1 per depth or until refusal | SERAS SOP #2016 | Biased – Based on bathymetric survey results |
| JWL-SED11-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP #2016 | Biased – Based on bathymetric survey results |
| JWL-SED11-B<br>JWL-SED11-C<br>JWL-SED11-D<br>JWL-SED11-E<br>JWL-SED11-F<br>JWL-SED11-G<br>JWL-SED11-H<br>JWL-SED11-I | Sediment | 6-12<br>12-24<br>24-36<br>36-48<br>48-60<br>60-72<br>72-84<br>84-96 | ICP Metals | Low | 1 per depth or until refusal | SERAS SOP #2016 | Biased – Based on bathymetric survey results |
| JWL-SED12-A | Sediment | 0-6 | TAL Metals | Low | 1 | SERAS SOP #2016 | Biased – Based on bathymetric survey results |

## QAPP Worksheet #18
## Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ID Number | Matrix | Depth ( inches) | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference | Rationale for Sampling Location |
|---|---|---|---|---|---|---|---|
| JWL-SED12-B | Sediment | 6-12 | ICP Metals | Low | 1 per depth or until refusal | SERAS SOP #2016 | Biased – Based on bathymetric survey results |
| JWL-SED12-C | | 12-24 | | | | | |
| JWL-SED12-D | | 24-36 | | | | | |
| JWL-SED12-E | | 36-48 | | | | | |
| JWL-SED12-F | | 48-60 | | | | | |
| JWL-SED12-G | | 60-72 | | | | | |
| JWL-SED12-H | | 72-84 | | | | | |
| JWL-SED12-I | | 84-96 | | | | | |

NA = not applicable

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #19
## Analytical SOP Requirements Table

| Matrix | Analytical Group | Concentration Level | Analytical and Preparation Method/SOP Reference [1] | Sample Volume | Containers (number, size, and type) | Preservation Requirements (chemical, temperature, light protected) | Maximum Holding Time (preparation/ analysis) |
|---|---|---|---|---|---|---|---|
| July 2011 | | | | | | | |
| Soil | Metals (Pb) | Low | SERAS SOP #1720 | 3-5 grams | (1) 4-oz. glass jar | Cool to 4°C | 6 months |
| Sediment | TAL Metals | Low | EPA R2 SOP# C-116/C-109/ C-127 | 1 g | (1) 8-oz. glass jar w/Teflon lined cap | Cool to 4°C | 5 months Hg- 28 days |
| Sediment | ICP Metals | Low | EPA R2 SOP# C-116/C-109 | 1 g | (1) 4-oz. glass jar | Cool to 4°C | 6 months |
| Water | ICP Metals | Low | EPA R2 SOP# C-116/C-109 or C-112 | 50 mL | (1) 1 liter poly | $HNO_3$ to pH<2; cool to 4°C | 6 months |
| Rinsate Blanks (from no dedicated soil/sediment equipment) if applicable | ICP Metals | Low | EPA R2 SOP# C-116/C-109 | 50 ml | (1) 1 liter poly | $HNO_3$ to pH<2;cool to 4°C | 6 months |
| Soil | ICP Metals | Low | EPA R2 SOP# C-116/C-109 | 0.5g | (1) 4 – oz. glass jar | Cool to 4°C | 6 months |
| **July-August 2012** | | | | | | | |
| Sediment | TAL Metals | Low | EPA R2 SOP# C-116/C-109/ C-127 | 1 g | (1) 8-oz. glass jar w/Teflon lined cap | Cool to 4°C | 6 months Hg- 28 days |
| Sediment | ICP Metals | Low | EPA R2 SOP# C-116/C-109 | 1 g | (1) 4-oz. glass jar | Cool to 4°C | 6 months |

## Analytical SOP Requirements Table

| Matrix | Analytical Group | Concentration Level | Analytical and Preparation Method/SOP Reference [1] | Sample Volume | Containers (number, size, and type) | Preservation Requirements (chemical, temperature, light protected) | Maximum Holding Time (preparation/ analysis) |
|---|---|---|---|---|---|---|---|
| Rinsate Blanks (from non-dedicated soil/sediment equipment) | ICP Metals | Low | EPA R2 SOP# C-116/C-109 | 50 ml | (1) 1 liter poly | $HNO_3$ to pH<2;cool to 4°C | 6 months |

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23)

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #20
## Field Quality Control Sample Summary Table

| Matrix | Analytical Group | Concentration Level | Analytical and Preparation SOP Reference[1] | No. of Samples | No. of Field Duplicate Pairs | Inorganic No. of MS[2] | No. of Field Blanks | No. of Equip. Blanks | No. of PT Samples | Total No. of Samples to Lab |
|---|---|---|---|---|---|---|---|---|---|---|
| **August 2012** | | | | | | | | | | |
| Water | ICP Metals | Low | EPA R2 SOP# C-116/C-109 | 32 (16 filtered, 16 unfiltered) | 2 | 2 | 1 per day (est. 5) | 1 per day (est. 7) | NA | 46 |
| Soil | Pb | Low | SERAS SOP #1720 | ≤144 | NA | NA | NA | NA | NA | 144 |
| Soil | ICP Metals | Low | EPA R2 SOP# C-116/C-109 | ≤32 | 2 | 2 | NA | NA | NA | 34 |
| Sediment | ICP Metals | Low | EPA R2 SOP# C-116/C-109 | ≤48 | 3 | 3 | NA | NA | NA | 51 |
| Sediment | TAL Metals | Low | EPA R2 SOP# C-116/C-109 | 6 | 1 | 1 | NA | NA | NA | 7 |
| **July – August 2012** | | | | | | | | | | |
| Sediment | ICP Metals | Low | EPA R2 SOP# C-116/C-109 | ≤48 | 3 | 3 | NA | 1 (rinsate blank) | NA | 52 |
| Sediment | TAL Metals | Low | EPA R2 SOP# C-116/C-109/ C-127 | 6 | 1 | 1 | NA | NA | NA | 7 |

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23)
[2]Assumes MS/MSD samples will be collected at a rate of 1 per 20 samples per property sampled by SERAS.

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #21
## Project Sampling SOP References Table

| Reference Number | Title, Revision Date and/or Number | Originating Organization | Equipment Type | Modified for Project Work? (Check if yes) | Comments |
|---|---|---|---|---|---|
| 2001 | General Field Sampling Guidelines | SERAS | General Sampling | No | |
| 2003 | Sample Storage, Preservation and Handling | SERAS | Sample Handling | No | |
| 2006 | Sampling Equipment Decontamination | SERAS | Ponar, or if any nondedicated sampling equipment is used. | No | |
| 2012 | Soil Sampling | SERAS | Aluminum pans, troughs | No | |
| 2016 | Sediment Sampling | SERAS | Aluminum pans, troughs, ponar | No | |
| 2013 | Surface Water Sampling | SERAS | Kemmerer bottle | No | |
| 2007 | Groundwater Sampling | SERAS | Portable pump and dedicated tubing. | No | |
| 2044 | Well Development | SERAS | Portable pump and dedicated tubing. | No | |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #22 – July 2011
## Field Equipment Calibration, Maintenance, Testing, and Inspection Table

| Field Equipment | Calibration Activity | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference |
|---|---|---|---|---|---|---|---|---|---|
| Niton XLt792YW Field Portable X-ray Fluorescence | Check target element response with reference standard | NA | Analyze reference standards | NA | With each use | Element results typically within ±20% of true values for concentrations 5x RL. For ERA SRM, %RSD ±20% | Repeat. If still fails, send in for factory service/calibration | XRF Analyst | 1720 |

[1]Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

## QAPP Worksheet #22 – August 2012
## Field Equipment Calibration, Maintenance, Testing, and Inspection Table

| Field Equipment | Calibration Activity | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference |
|---|---|---|---|---|---|---|---|---|---|
| Odom Echotrac CVM multi-frequency singel beam echo sounder | Bar Checks Twice Daily | Cleaning Transducer Daily | Hardware test prior to survey | Checking all connections | Daily | All QA/QC requirements met | Fix or replace any non functioning equipment. | PADOVER | ASI SOP SRV-004 |
| ODEC Stratbox system | Running test lines | Cleaning Transducer Daily | Hardware and software test prior to survey | Checking all connections | Daily | All QA/QC requirements met | Fix or replace any non functioning equipment. | PADOVER | ASI SOP SRV-010 |
| | | | | | | | | | |

[1]Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #23-July 2011
## Analytical SOP References Table

| Reference Number | Title, Revision Date, and/or Number | Definitive or Screening Data | Analytical Group | Instrument | Organization Performing Analysis | Modified for Project Work? |
|---|---|---|---|---|---|---|
| EPA R2 SOP #C-109 | Determination of Metals in Aqueous, TCLP Extracts, Soil/Sediment, Sludge, and Biological Tissue Samples by Inductively Coupled Plasma-Atomic Emission Spectrometry | Definitive | Metals | ICP | DESA | No |
| EPA R2 SOP #C-112 | Determination of Trace Elements in Aqueous, Soil/Sediment, Sludge, Waste Oil/Organic Solvents and Biological Tissue Samples by Inductively Coupled Plasma-Mass Spectrometry | Definitive | Metals | ICP-MS | DESA | No |
| EPA R2 SOP #C-116 | Preparation of Aqueous, TCLP Extracts, Soil/Sediment/Sludge/Solid, Biological Tissue and Other Matrices by Block Digestion | Definitive | Metals | NA | DESA | No |
| EPA R2 SOP #C-127 | Determination of Mercury in Aqueous, Soil/Sediment and Biological Tissue Matrices by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry | Definitive | Metals | CVAA | DESA | No |
| SERAS SOP #1720 | Operation of the Niton XLt792YW Field Portable X-ray Fluorescence Instrument, Rev. 1.0, 01/20/06 | Screening | Metals | FPXRF | ERT/SERAS Laboratory | No |

## QAPP Worksheet #23-July and August 2012
## Analytical SOP References Table

| Reference Number | Title, Revision Date, and/or Number | Definitive or Screening Data | Analytical Group | Instrument | Organization Performing Analysis | Modified for Project Work? |
|---|---|---|---|---|---|---|
| EPA R2 SOP #C-109 | Determination of Metals in Aqueous, TCLP Extracts, Soil/Sediment, Sludge, and Biological Tissue Samples by Inductively Coupled Plasma-Atomic Emission Spectrometry | Definitive | Metals | ICP | DESA | No |
| EPA R2 SOP #C-116 | Preparation of Aqueous, TCLP Extracts, Soil/Sediment/Sludge/Solid, Biological Tissue and Other Matrices by Block Digestion | Definitive | Metals | NA | DESA | No |
| EPA R2 SOP #C-127 | Determination of Mercury in Aqueous, Soil/Sediment and Biological Tissue Matrices by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry | Definitive | Metals | CVAA | DESA | No |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #24
## Analytical Instrument Calibration Table

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action (CA) | Person Responsible for CA | SOP Reference[1] |
|---|---|---|---|---|---|---|
| ICP-AES | See SOP C-109 | See SOP C-109 | See SOP C-109 | See SOP C-109 | Assigned Lab personnel | SOP C-109 |
| ICP-MS | See SOP C-112 | See SOP C-112 | See SOP C-112 | See SOP C-112 | Assigned Lab personnel | SOP C-112 |
| Mercury Analyzer | See SOP C-127 | See SOP C-127 | See SOP C-127 | See SOP C-127 | Assigned Lab personnel | SOP C-127 |

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23)

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #25
## Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

| Instrument/ Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference[1] |
|---|---|---|---|---|---|---|---|---|
| ICP-AES | As per instrument manufacturer's recommendations | As per instrument manufacturer's recommendations | As per instrument manufacturer's recommendations | As per instrument manufacturer's recommendations | As per instrument manufacturer's recommendations | Acceptable recalibration | ICP-AES Analyst | EPA R2 SOP # C-116/C-109 |
| ICP-MS | As per instrument manufacturer's recommendations | As per instrument manufacturer's recommendations | As per instrument manufacturers recommendations | As per instrument manufacturers recommendations | As per instrument manufacturer's recommendations | Acceptable recalibration | ICP-MS Analyst | EPA R2 SOP # C-116/C-109 |
| Mercury Analyzer | As per instrument manufacturer's recommendations | As per instrument manufacturer's recommendations | As per instrument manufacturers recommendations | As per instrument manufacturers recommendations | As per instrument manufacturer's recommendations | Acceptable recalibration | Hg Analyst | EPA R2 SOP #C-127 |

[1]Specify the appropriate reference letter or number from Analytical SOP References table (Worksheet #23)

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #26
## Sample Handling System

| |
|---|
| **SAMPLE COLLECTION, PACKAGING, AND SHIPMENT** |
| Sample Collection (Personnel/Organization): SERAS personnel |
| Sample Packaging (Personnel/Organization): SERAS personnel |
| Coordination of Shipment (Personnel/Organization): SERAS personnel |
| Type of Shipment/Carrier: Overnight delivery service or courier, personal delivery |
| **SAMPLE RECEIPT AND ANALYSIS** |
| Sample Receipt (Personnel/Organization): Sample Custodian at EPA R2 DESA |
| Sample Custody and Storage (Personnel/Organization): Sample Custodian |
| Sample Preparation (Personnel/Organization) EPA Region II DESA Inorganic Technicians/Chemists |
| Sample Determinative Analysis (Personnel/Organization: DESA Inorganic Technicians/Chemists |
| **SAMPLE ARCHIVING** |
| Field Sample Storage (No. of days from sample collection): Samples to be shipped within 24 hours of collection and arrive at laboratory within 24 hours (1 day) of shipment |
| Sample Extract/Digestate Storage (No. of days from extraction/digestion): As per analytical method |
| **SAMPLE DISPOSAL** |
| Personnel/Organization: SERAS |
| Number of Days from Analysis: In accordance with Laboratory's guidelines |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #27
## Sample Custody Requirements

| |
|---|
| Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Chain of custody records will be generated for all samples submitted for analysis per SERAS SOP #4005, *Chain of Custody Procedures*. Each sample will be individually labelled, then sealed with custody seals. Sample containers will be placed into Ziplock™ storage bags and then into a shipping cooler with the corresponding COC record. Samples will be shipped to the EPARegion 2 DESA laboratory via overnight deliveryservice or courier. |
| Laboratory Sample Custody Procedures (receipt of samples, archiving and disposal): A sample custodian at the laboratory will accept custody of the shipped samples, and check them for discrepancies, proper preservation, integrity, etc. If noted, issues will be forwarded to the laboratory manager for corrective action. The sample custodian will relinquish custody to the appropriate department for analysis. At this time, no samples will be archived at the laboratory. |
| Sample Identification Procedures: Each individual sample shall be given a unique ID. Sample identification will conform to SERAS SOP #2002, *Sample Documentation* and as specified in Worksheet #18 |
| Chain-of-custody Procedures: SERAS SOP #4005, *Chain of Custody Procedures* |

# QAPP Worksheet #28-1 – July 2011 and August 2012
# QC Samples Table

| | |
|---|---|
| **Matrix** | Sediment/Soil |
| **Analytical Group** | ICP – Metals |
| **Concentration Level** | Low/Medium (mg/kg) |
| **Sampling SOP(s)** | SERAS SOP #2016/2012 |
| **Analytical Method/SOP Reference** | EPA R2 DESA SOP #C-116/C-109 |
| **Sampler's Name** | C. Gussman/ SERAS personnel |
| **Field Sampling Organization** | SERAS |
| **Analytical Organization** | EPA DESA Laboratory |
| **No. of Sample Locations** | July 2011: Up to 54 (6 locations x 9 depths per location) for sediment<br>Up to 32 soil samples.<br>August 2012: Up to 54 (6 locations x 9 depths per location) for sediment |

| Lab QC Sample: | Frequency/Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator (DQI) | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| MB | 1 per ≤ 20 samples | < RL | If result > RL, all associated samples with a concentration ≤ 10 x should be re-prepped and reanalyzed or qualify data. | Laboratory Analyst | Accuracy/Bias (Contamination) | <RL |
| MS | 1 per ≤ 20 samples | 75-125%R* | Flag outliers | Laboratory Analyst | Accuracy | 75-125%R* |
| LCS/LCSD | 1 per ≤ 20 samples | ± 25% RPD** | Flag outliers | Laboratory Analyst | Precision | ± 25% RPD** |
| Serial Dilution | 1 per batch of 20 | ± 10% D | Flag outliers | Laboratory Analyst | Accuracy | ± 10% D |
| Interference Check Sample [ICP Analysis Only] | beginning, end and periodically during run (2 times every 8 hours) | Within ± 2 times CRQL of true value or ± 20% of true value, whichever is greater | Check calculations and instruments, reanalyze affected samples | Laboratory Analyst | Sensitivity | Within ± 2 times CRQL of true value or ± 20% of true value, whichever is greater |

| | | | | | | |
|---|---|---|---|---|---|---|
| Laboratory Control Sample | 1 per $\leq$ 20 samples | $\%R_{AVE} \pm$ 25% of true value. | Suspend analysis until source rectified; redigest and reanalyze affected samples | Laboratory Analyst | Accuracy/Bias | $\%R_{AVE} \pm$ 25% of true value. |

*except when the sample concentration is greater than 4 times the spike concentration, then disregard the recoveries; no data validation action taken
**Reference USEPA Region 2 SOP No. HW-2, Revision 13/Evaluation of Metals Data for CLP - (include absolute difference criteria)
**except when the sample and/or duplicate concentrations are less than 5 times the CRQL, then $\pm$ CRQL.
*** If the EPA LCS is unavailable, other EPA QC samples or other certified materials may be used. In such cases, control limits for the LCS must be documented and provided.

# QAPP Worksheet #28 – July and August 2012
# QC Samples Table

| | |
|---|---|
| **Matrix** | Soil |
| **Analytical Group** | Target Analyte List Inorganics – Metals |
| **Concentration Level** | Low/Medium (mg/kg) |
| **Sampling SOP(s)** | SERAS SOP #2016/2012 |
| **Analytical Method/SOP Reference** | ILM05.4 |
| **Sampler's Name** | C. Gussman/ SERAS personnel |
| **Field Sampling Organization** | SERAS |
| **Analytical Organization** | EPA CLP RAS Laboratory |
| **No. of Sample Locations** | August 2012: Up to 54 (6 locations x 9 depths per location) for sediment |

| Lab QC Sample: | Frequency/Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator (DQI) | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Preparation Blank | 1 per ≤ 20 samples | No constituent > CRQL | Suspend analysis until source rectified; redigest and reanalyze affected samples | EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician | Accuracy | No constituent > CRQL |
| Spike | 1 per ≤ 20 samples | 75-125%R* | Flag outliers | EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician | Accuracy | 75-125%R* |
| Duplicate | 1 per ≤ 20 samples | ± 20% RPD** | Flag outliers | EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician | Precision | ± 20% RPD** |
| Post-Digestion Spike | after any analyte (except Ag and Hg) fails spike %R | 75-125%R | Flag outliers | EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician | Accuracy | 75-125%R |
| Interference Check Sample [ICP Analysis Only] | beginning, end and periodically during run (2 times every 8 hours) | Within ± 2 times CRQL of true value or ± 20% of true value, whichever is greater | Check calculations and instruments, reanalyze affected samples | EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician | Sensitivity | Within ± 2 times CRQL of true value or ± 20% of true value, whichever is greater |

*except when the sample concentration is greater than 4 times the spike concentration, then disregrard the recoveries; no data validation action taken
**Reference USEPA Region 2 SOP No. HW-2, Revision 13/Evaluation of Metals Data for CLP - (include absolute difference criteria)
**except when the sample and/or duplicateconcentration is less than 5 times the CRQL, then ± CRQL.

## QAPP Worksheet #28 July and August 2012

## QC Samples Table

### (UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limit exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

| | |
|---|---|
| **Matrix** | Soil |
| **Analytical Group** | Target Analyte List Inorganics – Metals |
| **Concentration Level** | Low/Medium (mg/kg) |
| **Sampling SOP(s)** | SERAS SOP #2016/2012 |
| **Analytical Method/SOP Reference** | ILM05.4 |
| **Sampler's Name** | C. Gussman/ SERAS personnel |
| **Field Sampling Organization** | SERAS |
| **Analytical Organization** | EPA CLP RAS Laboratory |
| **No. of Sample Locations** | August 2012: Up to 54 (6 locations x 9 depths per location) for sediment |

| Lab QC Sample: | Frequency/Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator (DQI) | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Laboratory Control Sample | 1 per $\leq$ 20 samples | Control limits established by EPA* | Suspend analysis until source rectified; redigest and reanalyze affected samples | EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician | Accuracy | Control limits established by EPA* |

* If the EPA LCS is unavailable, other EPA QC samples or other certified materials may be used. In such cases, control limits for the LCS must be documnetd and provided.

# QAPP Worksheet #28-1 – July 2011
# QC Samples Table

| | |
|---|---|
| Matrix | Aqueous |
| Analytical Group | Metals |
| Concentration Level | Low |
| Sampling SOP | 2007/2013 |
| Analytical Method/ SOP Reference | C-109,C-112, C-116, C-127 |
| Sampler's Name | Chris Gussman/SERAS |
| Field Sampling Organization | SERAS |
| Analytical Organization | USEPA Region 2 Lab |
| No. of Sample Locations | |

| QC Sample: | Frequency/Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator (DQI) | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Tuning/System Stability(ICP-MS) | As per C-112 | Pass all the tune/stability criteria | Check Instrument Reanalyze, Retune | Lab personnel | Sensitivity | Pass all the tune/stability criteria |
| Initial Calibration Verification | Immediately following each calibration ,after every 10 samples and at the end of each analytical run | 90%-110% | Check Instrument, Reanalyze | Lab personnel | Accuracy | 90%-110% |
| Continuing Calibration Check Standard (Alternate check standard) | Every 10 samples and at the end of each analytical run | 80%-120% | Reanalyze, Qualify data | Lab personnel | Accuracy | 80%-120% |
| Initial Calibration Blank(ICB) | After ICV | < RL | Investigate source of contamination | Lab personnel | Sensitivity Contamination | < RL |
| Continuing Calibration Blank(CCB) | After every CCV | < RL | Investigate source of contamination | Lab personnel | Sensitivity Contamination | < RL |

| | | | | | | |
|---|---|---|---|---|---|---|
| Low Level Check Standard | At Beginning and end of each analytical run | ± 30% of the true value | Check Instrument, Re-calibrate | Lab personnel | Accuracy | ± 30% of the true value |
| Interference Check Sample( ICP-200.7) | At Beginning and end of each analytical run | < RL<br>Except Al ,Fe, Ca, K, Mg and Na | As per C-109 | Lab personnel | Precision | < RL<br>Except Al ,Fe, Ca, K, Mg and Na |
| Method blank | 1 per extraction batch of ≤ 20 samples | < RL | Investigate source of contamination | Lab personnel | Sensitivity Contamination | < RL |
| LCS/LFB | 2 per extraction batch of ≤ 20 samples | Limits: Average Recovery ± 20% aqueous, ± 25% Soil)<br>% RPD < 20( Aq),<br>% RPD <25(Soil) | Qualify data | Lab personnel | Accuracy/ Precision | Limits: Average Recovery ± 20% aqueous, ± 25% Solids)<br>% RPD < 20( Aq),<br>% RPD <25(Soil |
| Laboratory Matrix spikes | 1 per extraction batch of ≤ 20 samples | Limits ± 20% aqueous, ± 25% Soil) | Qualify data | Lab personnel | Accuracy | Limits ± 20% aqueous, ± 25% Soil) |
| Serial Dilution Test( ICP-200.7) | Matrix spike sample | RPD < 20 % | Qualify data | Lab personnel | Precision | RPD < 20 % |
| Internal Standards( ICP-MS 200.8) | Each sample, standard, blank | Range of 0.60-1.87 of the original response in the calibration blank | Check Instrument Analyze / Qualify data | Lab personnel | Quantitation | Range of 0.60-1.87 of the original response in the calibration blank |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #29
## Project Documents and Records Table

| Sample Collection Documents and Records | On-site Analysis Documents and Records | Off-site Analysis Documents and Records | Data Assessment Documents and Records | Other |
|---|---|---|---|---|
| Chain of custody records<br>Sample labels<br>Custody seals<br>Site Logbook<br>Field Change Form (if necessary) | XRF Results | Instrument run logs<br>Sample digestion logs<br>Preventative maintenance logs<br>Instrument printouts<br>Internal COC records<br>Temperature logs<br>Standard receipt logs<br>Standard prep logs<br>Data Reduction/Data Review records<br>Analytical Results | Data Validation Report | Technical Memorandum |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #30
## Analytical Services Table

| Matrix | Analytical Group | Concentration Level | Sample Location/ID Numbers | Analytical SOP | Data Package Turnaround Time | Laboratory/Organization (Name and Address, Contact Person and Telephone Number) | Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number |
|---|---|---|---|---|---|---|---|
| July 2011 | | | | | | | |
| Water | ICP Metals | Low | See Worksheet #18 | EPA R2 SOP# C-116/C-109 | 30 days | DESA Laboratory | NA |
| Soil | Pb | Low | See Worksheet #18 | SERAS SOP #1720 | NA | ERT/SERAS Laboratory Vinod Kansal (732-321-4252) | NA |
| Soil | ICP Metals | Low | See Worksheet #18 | EPA R2 SOP# C-116/C-109 | 30 days | DESA Laboratory | NA |
| Sediment | ICP Metals | Low | See Worksheet #18 | EPA R2 SOP# C-116/C-109 | 30 days | DESA Laboratory | NA |
| Sediment | TAL Metals | Low | See Worksheet #18 | EPA R2 SOP# C-116/C-109 | 30 days | DESA Laboratory | NA |
| July – August 2012 | | | | | | | |
| Water (Rinsate Blanks) | ICP Metals | Low | See Worksheet #18 | EPA R2 SOP# C-116/C-109 | 30 days | DESA Laboratory | NA |
| Sediment | ICP Metals | Low | See Worksheet #18 | EPA R2 SOP# C-116/C-109 | 30 days | DESA Laboratory | NA |
| Sediment | TAL Metals | Low | See Worksheet #18 | EPA R2 SOP# C-116/C-109 | 30 days | DESA Laboratory | NA |

NA – not applicable

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #31
## Planned Project Assessments Table

| Assessment Type | Frequency | Internal or External | Organization Performing Assessment | Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation) | Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation) | Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation) | Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation) |
|---|---|---|---|---|---|---|---|
| PT | Semiannually | External | NELAC | PT provider | Lab Personnel | Lab Personnel | Lab QA Officer |
| External NELAC Audit | Every two years | External | NELAC | Florida DOH | Lab QA Officer | Lab Personnel | Florida DOH |
| Internal Audit | Yearly | Internally | DESA Lab | Lab QA Officer | Lab Personnel | Lab Personnel | Lab QA Officer |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #32
## Assessment Findings and Corrective Action Responses

| Assessment Type | Nature of Deficiencies Documentation | Individual(s) Notified of Findings (Name, Title, Organization) | Timeframe of Notification | Nature of Corrective Action Response Documentation | Individual(s) Receiving Corrective Action Response (Name, Title, Org.) | Timeframe for Response |
|---|---|---|---|---|---|---|
| Proficiency Testing (PT) | Letter with PT failure indicated | Lab QA Officer | 30 days after the audit | Investigate the reason for the PT failure | Lab QA Officer | 45 days after the CA report |
| External NELAC Audit | Audit Report | Lab Management | 30 days after the audit | Investigate and have a corrective action plan for the deficiencies | Florida DOH | 30 days after receiving notification |
| Internal Lab Audit | Audit Report | Lab Management | 30 days after the audit | Investigate and have a corrective action plan for the deficiencies | Lab QA Officer | 30 days after the CA report |
| Peer Review | Directly on deliverable | Chris Gussman, Task Leader, SERAS | Prior to deliverable due date | Comments directly on deliverable | Christopher Gussman, Task Leader, SERAS | Prior to deliverable due date |
| Field observations/ Deviations from Work Plan | Logbook | Chris Gussman, Task Leader, SERAS | Immediately | Logbook | Christopher Gussman, Task Leader, SERAS | Within 24 hours of deviation. |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #33
## QA Management Reports Table

| Type of Report | Frequency (daily, weekly monthly, quarterly, annually, etc.) | Projected Delivery Date(s) | Person(s) Responsible for Report Preparation (Title and Organizational Affiliation) | Report Recipient(s) (Title and Organizational Affiliation) |
|---|---|---|---|---|
| Technical Report | Monthly | 10 th of the month following performance peiod | Task Leader/SERAS | ERT Project Officer and Work Assignment Manager |
| QA Report | Quarterly | February, May, August, November | QA/QC Officer/SERAS | ERT Quality Coordinator and ERT Project Officer |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #34
## Verification (Step I) Process Table

| Verification Input | Description | Internal/ External | Responsible for Verification (Name, Organization) |
|---|---|---|---|
| Chain of custody record | Reviewed byTask Leader in the field; original sent to laboratory | I/E | SERAS Task Leader, Christopher Gussman, EPA R2 DESA Lab |
| Laboratory analytical data package | Data packages will be reviewed/verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. | E | EPA Region 2 DESA Lab |
| Technical Memorandum Report | Verified that transcription errors are not present | I | Peer review team |
| Completeness Check | Review of Planning Documents, Analytical Data Package, Sampling Documents and External reports, as applicable, using the UFP-QAPP Checklist | I | SERAS Task Leader, Christopher Gussman, |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #35
## Validation (Steps IIa and IIb) Process Table

| Step IIa/IIb | Validation Input | Description | Responsible for Validation (Name, Organization) |
|---|---|---|---|
| IIa | SOPs | Ensure that the sampling methods/procedures outlined in QAPP were followed, and that any deviations were noted/approved. | SERAS Task Leader & ERT WAM |
| IIb | SOPs | Determine potential impacts from noted/approved deviations, in regard to PQOs. | EPA Data Validation Personnel |
| IIa | Chains of custody | Examine COC forms against QAPP and laboratory contract requirements (e.g., analytical methods, sample identification, etc.). | SERAS Task Leader, EPA Data Validation Personnel, |
| IIa | Laboratory data package | Examine packages against QAPP and laboratory contract requirements, and against COC forms (e.g., holding times, sample handling, analytical methods, sample identification, data qualifiers, QC samples, etc.). | Data Validation Personnel. |
| IIb | Laboratory data package | Determine potential impacts from noted/approved deviations, in regard to PQOs. Examples include PQLs and QC sample limits (precision/accuracy). | EPA Data Validation Personnel, EPA Region 2 |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #36
## Validation (Steps IIa and IIb) Summary Table

| Step IIa/IIb | Matrix | Analytical Group | Concentration Level | Validation Criteria | Data Validator (title and organizational affiliation) |
|---|---|---|---|---|---|
| IIb | Sediment/ Aqueous/Soil | TAL Metals, ICP Metals | Low | EPA R2 Data Validation Guidelines | EPA Region 2 Data Validation Personnel |

☐ Worksheet Not Applicable (State Reason)

## QAPP Worksheet #37
## Usability Assessment

| |
|---|
| Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: |
| Describe the evaluative procedures used to assess overall measurement error associated with the project: |
| Identify the personnel responsible for performing the usability assessment: |
| Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: |

-**Precision:** Results of laboratory duplicates will be assessed during data validation and data will be qualified according to the data validation procedures cited in worksheet# 36. Field duplicates will be assessed during by matrix using the RPD for each pair of results above the QL for the performed analyses. RPD acceptance criteria, presented in worksheet #12, will be used to access field sampling precision. Absolute difference will be used for low results as described in worksheet # #28. A discussion summarizing the results of laboratory and field precision and any limitations on the use of the data will be described.

-**Accuracy/Bias Contamination:** Results for all laboratory blanks will be assessed as part of the data validation. During the data validation process, the validating personnel will qualify the data following the procedures described on worksheet #36. A discussion summarizing the results of the laboratory accuracy and bias based on contamination will be presented and any limitations on the use of the data will be described.

-**Overall Accuracy/Bias**: The results of instrument calibration and matrix spike recoveries will be reviewed and data will be qualified according to the data validation procedures cited on worksheet #36. A discussion summarizing the results of laboratory accuracy and any limitations on the use of the data will be described.

-**Sensitivity:** Data results will be compared to criteria provided in worksheet #15. A discussion summarizing any conclusions about the sensitivity of the analyses will be presented and any limitations on the use of the data will be described.

-**Representativeness:** Data representativeness will be assessed by collecting field replicate samples. The field replicates are by definition equally representative of a given point and space and time. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. Therefore, data representativeness will be satisfied by ensuring that:

The sampling program is followed according to:

U.S. EPA (Environmental Protection Agency). October 1989. *Region II CERCLA Quality Assurance Manual.* Final Copy, Revision 1. Division of Environmental

Services and Assessment, Edison, NJ; and

U.S. EPA (Environmental Protection Agency). December 1995. *Superfund Program Representative Sampling Guidance.* OSWER Directive 9360.4-10. Interim Final. EPA/540/R-95/141. Office of Emergency and Remedial Response (OERR). Washington, D.C.

U.S. EPA Environmental Response Team. *Standard Operating Procedure 2016, Sediment Sampling, 2012, Soil Sampling, 2013, Surface Water Sampling and 2007, Groundwater Sampling.*

-**Comparability:** To ensure data comparability, sampling and analysis for all samples will be performed using standardized analytical methods and adherence to the quality control procedures outlined in the methods and this QAPP. Therefore, the data will be comparable.

-**Reconciliation:** The PQOs presented in worksheet #11 will be examined against the data quality to determine if the objectives were met. This examination will include a combined overall assessment of the results of each analysis pertinent to an objective. Each analysis will first be evaluated separately in terms of major impacts observed from data validation, data quality indicators, and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. Based on the quality determined, the usability of the data for each analysis will be determined. Based on the combined usability of the data from all analyses for an objective, it will be determined if the PQOs were met and whether project goals are being achieved. Conclusions will be drawn and any limitations on the usability of the data will be described.

-**Completeness**: 1. To calculate field precision: $RPD = 100 \times \left( \frac{|X_1 - X_2|}{(X_1 + X_2)/2} \right)$ where X1 and X2 are the reported concentrations for each duplicate or replicate.

2. Calculate completeness: Data completeness will be expressed as the percentage of valid data obtained from measurement system. In other words, every well or location that was initially intended to be sampled, was sampled. For data to be considered valid, it must meet all the acceptable criteria including accuracy and precision, as well as any other criteria specified by the analytical method used. Therefore, all data points critical to the sampling program in terms of completeness will be 100% validated by USEPA Region II DESA/LB according to the appropriate and current US EPA Region 2 Data Validation SOPs G-26. With 100% validation, the rationale for considering data points non-critical is not required.

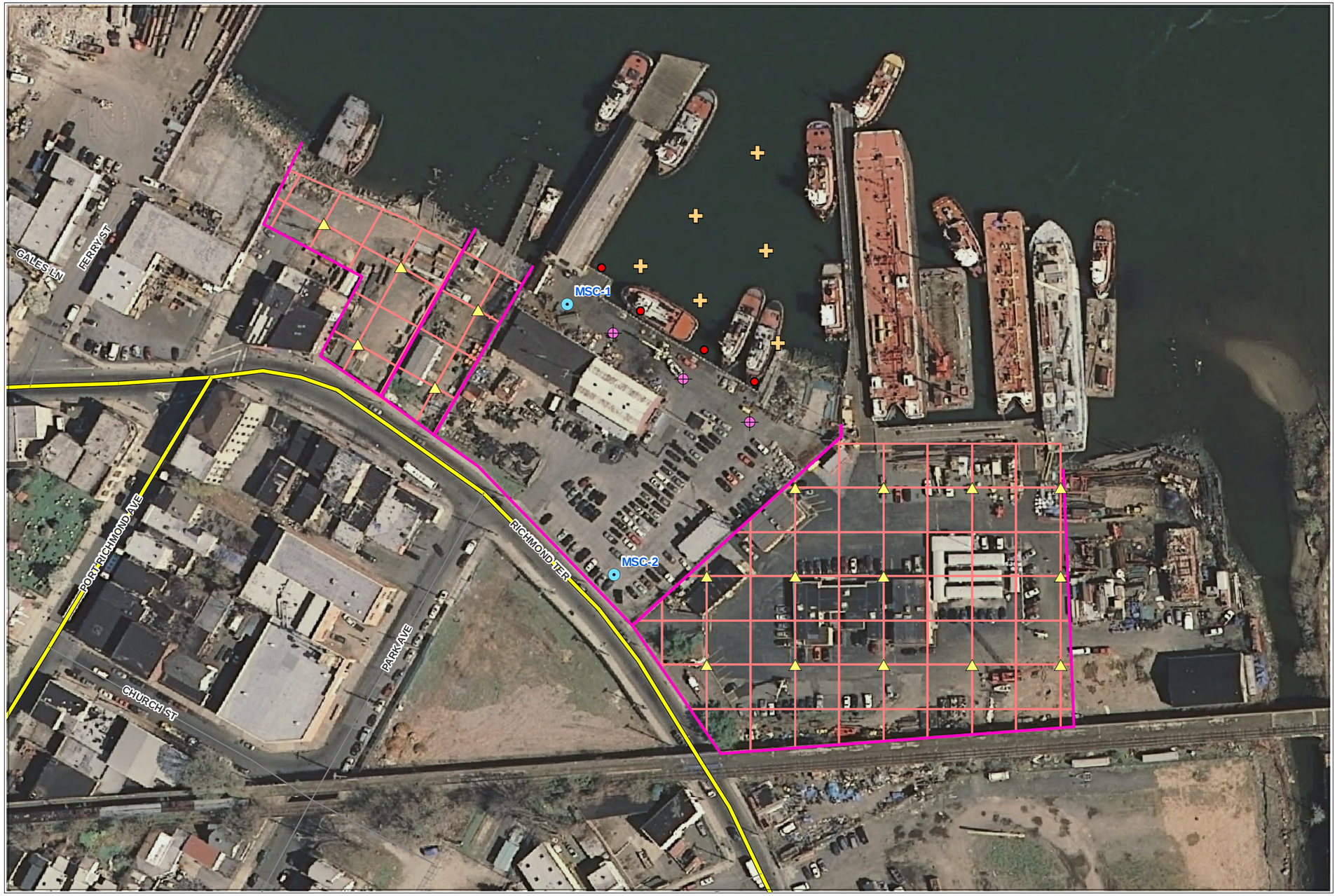
**Describe the evaluative procedures used to assess overall measurement error associated with the project:**

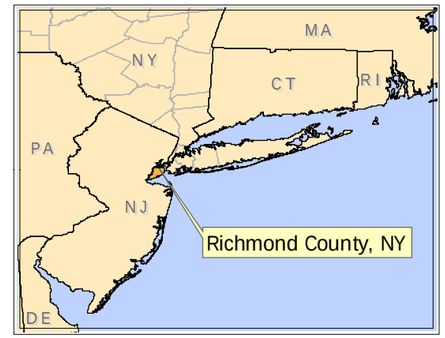
EPA Region 2 DESA-HWSB will determine if quality control data is within specification through validation process IIb.

**Identify the personnel responsible for performing the usability assessment:**

U.S. EPA HWSB-SST

FIGURE 1
Site Sampling Areas
Jewett White Lead Site
August 2012





0 2,500 Feet

Legend

- Existing Monitor Well
- Approximate Location of Vibracoring
- Approximate Location of Well Installation
- Approximate Location of Water sample *(Surface, Middle, Bottom)*
- Specific Soil Sample Location *(Additional Locations may be selected)*
- Property Line
- General Area of Geoprobe Samples

Map create using 2007 color orthophotography from NJGIN.

Map Creation Date: 10 May 2011

Coordinate system: New York State Plane
FIPS: 3104
Datum: NAD83
Units: Feet

120 0 120 Feet

N
W E
S
Magnetic North

D R A F T

U.S EPA Environmental Response Team
Scientific Engineering Response and Analytical Services
EP-W-09-031
W.A.# 0-138

Figure 1
Jewett White Lead Site Sampling Areas
Jewett White Lead Site
Staten Island, New York

Data: g:\arcviewprojects\SERAS01\00-138
MXD file: g:\arcinfoprojects\SERAS01\SER00138_JewettWhiteLead\138_Proposed_Site_Sampling_Area_f1

# APPENDIX A
EPA Region 2 DESA Laboratory SOPs
Jewett White Lead Site
August 2012

EPA

# STANDARD OPERATING PROCEDURE

# DETERMINATION OF METALS IN AQUEOUS, TCLP EXTRACTS, SOIL/SEDIMENT, SLUDGE, AND BIOLOGICAL TISSUE SAMPLES BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY

*Signature and Title*

Prepared by: Renée Lettieri — 3-12-09
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Approved by: [signature] — 3/13/09
John R. Bourbon, Acting Chief, Laboratory Branch — Date

*Annual Review*

Reviewed by: _____ _____
Signature — Date

Reviewed by: _____ _____
Signature — Date

**U.S. ENVIRONMENTAL PROTECTION AGENCY**
**REGION 2**
**DIVISION OF ENVIRONMENTAL SCIENCE AND ASSESSMENT**
**LABORATORY BRANCH**

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## TABLE OF CONTENTS

# STANDARD OPERATING PROCEDURE

# DETERMINATION OF METALS IN AQUEOUS, TCLP EXTRACT, SOIL/SEDIMENT, SLUDGE, AND BIOLOGICAL TISSUE MATRICES BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY

## 1. Scope and Application

1.1 This SOP is applicable to the analysis of environmental samples, including aqueous, TCLP extract, soil/sediment, sludge, biological tissue, and, for the determination of the following metals:

Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, and Zn

B, Sn, Si and Sr are not included in the analysis by iCAP 6300.

Note - This SOP is not applicable to the preparation and analysis of drinking water compliance monitoring samples. The procedure for the preparation and analysis of drinking water compliance monitoring samples by ICP-AES is detailed in Laboratory SOP DW-5.

Waste oil and organic solvents may be analyzed by this method following a suitable sample preparation procedure.

1.2 All analysts must satisfactorily perform an initial demonstration of capability (DOC) by meeting the method performance criteria in Sec. 13.1 prior to performing sample analysis using this SOP.

1.3 The standard reporting limits for both aqueous and non-aqueous samples are listed in Table 2.

1.4 This SOP is based on EPA Method 200.7, Revision 4.4.

## 2. Summary of Method

2.1 Environmental samples, e.g., aqueous, TCLP extracts, soil/sediment, sludges and biological tissue, are digested in a mixture of acids, according to the procedures described in U. S. Environmental Protection Agency, Region 2, SOP C-116 "Preparation of Aqueous, TCLP Extracts, Soil/Sediment/Sludge, and Biological Tissue Matrices by Block Digestion."

2.2 The analysis described in this method involves multi-element determinations by ICAP-AES using the Thermo IRIS Intrepid II ICAP or the Thermo iCAP 6300 Duo. These instruments measure characteristic atomic-line emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element specific emission spectra are produced by a radio-frequency inductively coupled argon plasma. The spectra are dispersed and the intensities of the line spectra are monitored at specific wavelengths using a Charge Injection Device (CID). The output from the detector is processed and controlled by a computer system.

A background correction technique is required to compensate for background contribution to the determination of the analytes. Background must be measured adjacent to the analyte wavelength during analysis. Various interferences must be considered and addressed appropriately.

## 3. Definitions

See **SOP G-15** for definitions.

## 4. Interferences

4.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

4.1.1 Spectral Interferences can be categorized as

1) overlap of a spectral line from another element;
2) unresolved overlap of molecular band spectra;
3) background contribution from continuous or recombination phenomena; and
4) background contribution from stray light from the line emission of high concentration elements.

The first of these effects can be compensated for by utilizing a computer correction of the raw data, requiring the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated for by a background correction adjacent to the analyte line. In addition, users of simultaneous multi element instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array. For this purpose, linear relationships between concentration and intensity for the analytes and the interferences must be demonstrated over the range of interest.

4.1.2 Physical Interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface

tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample. Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow-rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem. This problem can also be alleviated by using a Bergener nebulizer instead of a Meinhardt nebulizer. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

4.1.3 Chemical Interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), buffering of the sample and matrix matching. These types of interferences can be highly dependent on matrix type and specific analyte element.

4.2 Generally, whenever a new or unusual sample matrix is encountered, a series of tests on the matrix-type are performed, e.g., background check of the sample, sample overlay with standards, etc., prior to analyzing samples associated with that matrix. If the problems associated with the new matrix cannot be overcome, the sample must either be diluted appropriately (and the Reporting Limit raised accordingly) or analyzed by an acceptable alternative method.

## 5. Safety

The toxicity and carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices, e.g. wear proper protective equipment, safety glasses, gloves, lab coat and working in side hoods whenever possible. Refer to Edison Facility Safety Manual Region II (available on the Region II Intranet), Part 2 – Laboratory Safety and Appendices 13/13A - Chemical Hygiene Plan for specific guidelines.

## 6. Apparatus and Material

6.1. Inductively Coupled Argon Plasma Spectrometer:

6.1.1 Thermo IRIS Intrepid II or Thermo iCAP 6300 Duo, each computer controlled and equipped with a radio frequency generator and a variable speed peristaltic pump which is used to deliver both standards and samples to the nebulizer.

6.1.2 High purity (99.99%) liquid argon.

6.1.3 Computer controlled mass flow controllers which regulate the argon flow rates.
6.1.4 Autosampler, - as supplied by Thermo, Cetac or equivalent.

6.2. Balance which has the capability to measure to at least 0.01g.

6.3 Calibrated automatic pipets with disposable tips.

6.4. Miscellaneous laboratory glassware and plastic ware.

## 7. Reagents and Solutions

7.1 Reagents - All reagents must be of high purity and suitable for trace metals analysis.

7.1.1 Hydrochloric acid, concentrated - HCl (GFS HCl, 37% Reagent ACS or equivalent)

7.1.2 Nitric acid, concentrated - $HNO_3$ (GFS $HNO_3$, Redistilled or equivalent)

7.1.3 Reagent grade water – ASTM Type I Water

7.2 Solutions – All purchased standard solutions are required to be manufactured under UL ISO 9001 Quality Assurance Program. Refer to Table 1 for standard solutions preparation summary. Solutions are prepared using 2%HNO3 and 5% HCl.

7.2.1 Calibration Stock Standard Solutions - Claritas Custom Standards manufactured by Spex CertiPrep or equivalent. These solutions are usually used with the IRIS Intrepid II:

7.2.1.1 Calibration Standard 1 - SPEX CertiPrep Custom Claritas Standard (250 ppm of Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Sr, Ti, Tl, V, and Zn) or equivalent.

7.2.1.2 Calibration Standard 2 - SPEX CertiPrep Custom Claritas Standard (250 ppm of Al, Ca, Fe, Mg, K, Na and Si) or equivalent.

7.2.2 Calibration Stock Standard Solutions - The following solutions, available from Absolute Standards or equivalent, and are usually used with the iCAP 6300:

7.2.2.1 Calibration Stock A - Absolute ICP Mix 1 (500 ppm) – 17 custom mixed analytes containing As, Ba, Be, Cd, co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Ti, TL, V and Zn.

7.2.2.2 Calibration Stock B – Absolute Metals Mix (5,000ppm) mixed salts containing Al, Ca, Fe, K, Mg and Na.

7.2.2.3 Calibration Stock C - Absolute Silver (1000 ppm) single analyte containing Ag

only.

7.2.3 Single element standards for preparation of RL and IEC solutions. These may also be used to prepare Calibration Standards.

7.2.3.1 1,000 ppm Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Sr, Ti, Tl, V, and Zn. Available from GFS, Absolute or equivalent.

7.2.3.2 10,000 ppm Al, Ca, Fe, Mg, K, Na and Si. Available from GFS, Absolute or equivalent.

7.2.4 Working Calibration Solutions

7.2.4.1 The IRIS usually utilizes:

| | |
|---|---|
| High Standard | 10,000 µg/L for Al, Ca, Fe, K, Mg, Na and Si<br>1,000 µg/L for elements Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Sr, Ti, Tl, V, and Zn |
| Mid Standard | 5,000 µg/L for Al, Ca, Fe, K, Mg, Na and Si<br>500 µg/L for elements Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Sr, Ti, Tl, V, and Zn |

A single mixed standard or any other suitable combinations of standards/concentrations/ elements may also be used to standardize/calibrate the IRIS.

7.2.4.2 The Thermo iCAP 6300 usually utilizes:

| | |
|---|---|
| High Standard | 10,000 µg/L for Al, Ca, Fe, K, Mg, ,Na, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Ti, Tl, V, Zn, and 1000 µg/L for Ag |
| Mid Standard | 5,000 µg/L for Al, Ca, Fe, K, Mg, Na,<br>As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Ti, Tl,<br>V, Zn, and 500 µg/L for Ag |

A single mixed standard or any other suitable combinations of standards/ concentrations/elements may also be used to standardize/calibrate the iCAP 6300

B, Sn, Sr and Si are not usually included in analysis by iCAP 6300

7.2.5 Blanks – Four types of blanks are required for the analysis:

7.2.5.1 The calibration blank is prepared by adding $HNO_3$ and HCl to reagent grade water to the same concentrations used for the calibration standard solution. This calibration blank is used in establishing the analytical curve.

7.2.5.2 The rinse blank is prepared by adding $HNO_3$ and HCl to reagent grade water to the same concentration as used in the calibration blank. A controlled flush time with the rinse blank solution is used to flush the instrument uptake system and nebulizer between /standards/check solutions/samples to reduce/eliminate memory and carryover interferences.

7.2.5.3 The initial calibration blank/continuing calibration blank (ICB and CCB) are prepared by adding $HNO_3$ and HCl to reagent grade water to the same concentration as used in the calibration blank. The ICB/CCB is run after the calibration check standards to assess carryover.

7.2.5.4 Laboratory reagent blank (LRB)/Prep Blank (PB) - must contain all the reagents in the same volumes as used in digesting the samples. The LRB/PB must be carried through the same preparation scheme as the samples including digestion, if applicable. An LRB/PB is used to assess possible contamination from the sample preparation procedure and to assess spectral background.

7.2.6 Initial Calibration Verification/Continuing Calibration Verification Solution (ICV/CCV) – These verification standard solutions are used to initially and periodically to verify instrument performance during analysis. The ICV/CCV stocks must be obtained from a source different from the calibration stock standard solutions (different vendor or lot number) and prepared in the same acid mixture as the calibration standards.

7.2.6.1 IRIS - The concentration of the analytes in the ICV/CCV solution is usually 200 µg/L for elements: Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Sr, Ti, Tl, V and 5,000 µg/L for minerals: Al, Ca, Fe, K, Mg, Na and Si. (The concentration of the ICV/CCV solution may be varied to accommodate special requirements for non routine projects. Acceptance criteria will remain the same.)

7.2.6.1.1 Claritas Custom Standard - ICV1, 250ppm ( Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Sr, Ti, Tl, V, and Zn) or equivalent

7.2.6.1.2 Claritas Custom Standard - ICV2, 250ppm (Al, Ca, Fe, K, Mg, Na, Si) or equivalent.

7.2.6.2 iCAP 6300 The concentration of the analytes in the ICV/CCV solution is 100 µg/L for elements: Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Ti, Tl, V and 2,000 µg/L for minerals: Al, Ca, Fe, K, Mg, and Na.

7.2.6.2.1 Calibration Stock A - Absolute ICP Mix 1 (100 ppm) – 17 custom mixed analytes containing As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Ti, TL, V and Zn.

7.2.6.2.2 Calibration Stock B – Absolute Metals Mix (5,000ppm) mixed salts

containing Al, Ca, Fe, K, Mg and Na.

7.2.6.2.3 Calibration Stock C - Absolute Silver (1000 ppm) single analyte containing Ag only.

7.2.6.2.4 B, Sn, Sr and Si are not usually included in analysis by iCAP 6300. When required, B, Sn and Sr are included in the ICV/CCV at 100 µg/L and Si at 2,000 µg/L.

7.2.7 Low Level Check (RL or 2 RL) – The low level checks are used to initially and periodically verify instrument performance at lower concentration levels. The concentration of the analytes is set at the reporting limit (RL) and at twice the reporting limit (2 RL) for each element.

7.2.8 Internal Standard Solutions - The normal calibration procedure for arc/spark involves the use of an internal standard. An element not found in the matrix being analyzed is added to each standard and each sample. Should the volume of aspirated sample change, a corresponding intensity change will occur for all elements. Since the ratio remains constant, the possible error is eliminated.

Dilute Internal Standard Stock Solutions (Yttrium and Cesium, each 10,000 ppm or equivalent) to the following concentrations using 2% $HNO_3$ and 5% HCl:

7.2.8.1 IRIS: - 10ppm Y

7.2.8.2 iCAP 6300 -: 2,000 ppm Cs, 5 ppm Y

7.2.9 Auto Peak Adjust - use the high standard solution and follow the Auto Peak Adjust command on the instrument.

7.2.10 Inter-Element Correction (IEC) Solution - When inter-element corrections are applied, a spectral interference check solution is needed which contains concentrations of the interfering elements at levels that will provide an adequate test of the correction factors. The IEC solution is prepared by diluting individual standard solutions: Al, Ca, Fe, Mg and Na, each 10,000 ppm, to a final concentration of 300 ppm with 2% $HNO_3$/5% HCl blank solution.

## 8. Sample Collection, Preservation, Storage and Holding Time

8.1 Sample Collection - Samples must be collected in plastic or glass containers.

8.2 Preservation and Storage

8.2.1 Aqueous samples - the samples are preserved using concentrated $HNO_3$. The preservation is performed either a) in the field at the time of collection, or b) in the Laboratory upon receipt (within five business days). If the samples are preserved in the Laboratory, they must be held for sixteen hours after acidification and then verified to a pH<2 prior to sample processing. If the sample pH is verified to be pH>2 after the sixteen hours, additional $HNO_3$ must be added and the sample held for an additional sixteen hours until verified to a pH<2. The samples are stored at room temperature.

8.2.2 Soil/Sediment/Sludge samples - these samples are preserved in a refrigerator at ≤4°C. Alternatively, the samples may be stored at ≤-20°C in a freezer.

8.2.3 Biological Tissue samples - The samples are stored at ≤-20°C in a freezer.

8.2.4 Drum Samples – There is no temperature requirement for these samples.

8.3 Holding time

8.3.1 Aqueous samples must be prepared and analyzed within six months of collection.

8.3.2 Soil/Sediment/Sludge samples must be digested and analyzed within six months of collection.

Note: If soil/sediment samples are stored at ≤-20°C, the holding time is extended. The samples must be prepared within 12 months of collection and analyzed within 6 months of digestion.

8.3.3 Biological Tissue samples must be digested within 12 months of collection and analyzed within 6 months of digestion.

8.3.4 Drum Samples - a holding time is not established for the digestion or analysis of these samples.

## 9. Sample Preparation

All Environmental samples, e.g., aqueous, soil/sediment, and biological tissue, including NPDES wastewater compliance monitoring samples, are digested in a mixture of acids using the procedures described in SOP Number C-116 "Digestion of Metals Aqueous, TCLP Extracts Soil/Sediment, Sludge, and Biological Tissue Matrices by Block Digestion".

## 10. Instrument Operating Conditions

The analyst should follow the manufacturer's instructions for each instrument unless other

conditions provide better performance.

10.1 IRIS - Before lighting the plasma, make sure the following settings are in place:

RF power - 1150W
Auxiliary Gas – 0.75 L/min
Nebulizer Flow Rate – 0.75 L/min
Pump Rate – (Flush and Analysis) – 120 rpm

Note: Setting ranges are specified at safe or near optimum values, but may be adjusted as necessary.

Allow the plasma to become stable (about 45 minutes).

10.2 iCAP 6300 - Before lighting the plasma, make sure the following settings are in place:

Recirculating Chiller – 15-25 °C, 5L/minute flow
RF power – 1100 – 1200 W
Regulated Argon Pressure – 90-100 psi
Auxiliary Gas – 0.5 L/min
Nebulizer Flow Rate – 0.2 – 0.3 MPa (manually set)
Flush Pump Rate – 60 – 80 rpm
Analysis Pump Rate – 35 – 45 rpm

Note: Setting ranges are specified at safe or near optimum values, but may be adjusted as necessary.

Allow the plasma to become stable (about 45 minutes).

## 11. Sample Analysis

11.1 Configure the instrument settings to those in Section 10.

11.2 Fill in the sample ID file.

11.3 Calibrate the instrument using High and Mid Standard mixed calibration solutions and the calibration blank solution. The average of three readings is to be used. Adequately flush the system with the rinse blank solution between each determination... (A single mixed standard or any other suitable combinations of standards/concentrations/elements may also be used to standardize/calibrate the IRIS or iCAP 6300.)

11.4 After the completion of the initial requirements, samples should be analyzed in the same operational manner used in the standardization routine with a sample flush period being used

between all sample solutions, LFBs/LCSs-Aqueous, LFMs/MSs, and check solutions.

11.5 During the analysis of samples, the laboratory must comply with the required quality control in Sections 14. Only for the "direct analysis" of drinking water is the sample digestion step of the LRB/PB, LFB/LCS-Aqueous, and LFM/MS not required.

11.6 Sample analysis consists of the following:

Calibration Blank
Mid and High Mixed Standard Solutions (or Mixed Standard)
IPC/ICV
IPB/ICB (Calibration Blank solution)
Low Check Standard - RL and 2 RL
IEC
LRB/PB
LFBs/LCSs
Samples
LFM/MS and SD
IPC/CCV -must be analyzed at a minimum of every 10 samples
IPB/CCB - must be analyzed at a minimum of every 10 samples
Low Check Standard - RL and 2 RL
IEC

Note: The IPC/CCV and IPB/CCB must be analyzed at a minimum of every 10 analyses and at the end of each analysis run.

11.7 Determined sample analyte concentrations that are 90% or more of the upper limit of the analyte LDR must be diluted with reagent grade water that has been acidified in the same manner as the calibration blank and reanalyzed

11.8 Report Data as directed in Section 12.

## 12. Data Analysis and Calculations – IRIS and iCAP 6300

12.1 Aqueous Samples – Undigested

All dilution factors required as a result of dilutions made during analysis are applied at the instrument. Therefore, all of the aqueous sample results generated from the analysis (in µg/L), can be reported directly from the instrument. All results are reported to two significant figures and, in most cases, are reported down to the standard reporting limits listed in Table 2 or 3.

12.2 Aqueous Samples – Digested

All dilution factors required as a result of dilutions made during analysis are applied at the instrument. Therefore, all of the aqueous sample results, generated from the analysis (in µg/L), can be reported directly from the instrument. All results are reported to two significant figures and, in most cases, are reported down to the standard reporting limits listed in Table 2.

12.3 Non-Aqueous Samples

All dilution factors required as a result of dilutions made during analysis are applied at the instrument. Therefore, all of the results, generated from the analysis (in µg/L), can be used directly from the instrument. These "µg/L" results must then be converted to "mg/Kg" results. The µg/L result is multiplied by the final digestate volume in Liters, usually 0.050L, and divided by the sample mass in grams, usually 0.50g (the specific sample volume and mass are recorded in the metals sample preparation log book). For dry weight calculation, the mg/Kg results must be divided by the decimal version of the percent solids, e.g., 90% is 0.90. Refer to SOP G-23 for Percent Dry Solids.

$$Sample\,\mathrm{Re}sult, ug/g \;\; or \;\; mg/Kg, (dry\ weight\ basis = \frac{ug/L \;\; x \;\; V}{W \;\; x \;\; (\%\ Solids/100)}$$

where µg/L = Instrument reading (average of three replicates)
V = Final sample volume in liters (e.g. 0.050L)
W = Weight of wet sample in grams (e.g. 0.50g)

All mg/Kg results are reported to two significant figures and, in most cases, are reported down to the standard reporting limits listed in Table 2., adjusted for percent solids correction for dry weight basis.

## 13. Method Performance

An initial demonstration of capability (DOC) must be performed each time there is a significant change in the chemistry of the method, a major modification to an existing instrument, or a new instrument is installed. A DOC is performed by each analyst designated to analyze samples using this method. An annual check must subsequently be performed and documented for each analyst using this method.

13.1 Accuracy and Precision

13.1.1 Initial Demonstration of Capability

An initial demonstration of capability study must be conducted for this method for each analyst using this method. The study consists of the analysis of four standards or LCS's which are from a source independent of the standard curve. The results of the standards must be within the acceptance criteria supplied by the manufacturer or within 10% if none are specified. The % RSD should be within 20%. The results of the accuracy and precision study (true value, % recovery, standard deviation and %RSD) are maintained by the Quality Assurance Officer for each analyst and are located in the Central Branch File.

13.1.2 Continuing Demonstration of Capability

An annual continuing demonstration of capability study must be performed and documented. It may consist of either successfully analyzing a PT sample or analyzing 2 sets of LCS standards to within control limits as stated in section 13.1.1. The results of the continuing accuracy and precision study (true value, % recovery, standard deviation and % RSD or final report from the PT provider) are maintained by the Quality Assurance Officer for each analyst and are located in the Central Branch File.

13.2 Method Detection Limit (MDL)

An MDL Study must be conducted for this method. The study is based on the requirements listed in 40 CFR Part 136 Appendix B. Specific procedures for conducting an MDL study can be found in SOP # G-8. The MDL Study comprises the analysis of seven reagent grade water samples fortified at a level between 2-3x the detection limit. The results of the MDL determination (true value, average concentration, standard deviation and calculated MDL) are maintained by the Quality Assurance Officer for each method and are located in the Central Branch File.

13.3 Linear Dynamic Range (LDR)

The LDR must be determined by generating a normal linear calibration curve followed by the analysis of successively higher standard solutions. The results of these standard solutions are used to calculate % recovery. This is conducted until the % recovery falls below 90%. The last standard that had a % recovery of at least 90%is identified as the LDR limit.

The LDR should be verified annually or whenever in the judgement of the analyst, there is a change in analytical performance due to a significant change in instrument hardware or operating conditions

The results of the LDR Study are maintained in a file next to the instrument. The LDR results must be below or equal to that listed as the upper range in EPA Method 200.7.

13.4 Limit of Quantitation (LOQ)

The Laboratory performs a Limit of Quantitation (LOQ) study on an annual basis for analytes associated with chemistry methods. The validity of LOQ is confirmed by successful analysis of a Laboratory Fortified Blank (LFB) at approximately 2X the reporting limit. The acceptance criteria for each analyte is ± 30% of the true value. After this study is completed, it is reviewed and approved by the Laboratory Management. A summary of all LOQ study performance is maintained in the Laboratory's Central File.

## 14. Quality Control

If QC criteria provided in this method are not achieved, then corrective action(s) should be implemented. This may include sample re-analysis as determined by existing laboratory policy and/or in consult with lab management and QAO.

### 14.1 Calibration Curve

Acceptance Criteria –

The IRIS and iCAP6300 are calibrated using a Mid and High standard and a calibration blank. The correlation coefficient for each analyte of interest must be ≥ 0.995.

A single mixed standard or any other suitable combinations of standards/concentrations/ individual elements may be used to standardize/calibrate the IRIS. Acceptance criteria remain the same.

After standardization, the ICV and ICB are used to determine acceptance.

Corrective Action - If the results of the ICV or ICB are unacceptable, analysis must be discontinued, the cause determined and/or in the case of drift the instrument re-calibrated.

### 14.2 Initial Calibration Verification (ICV).

Acceptance Criteria - Analyze the ICV solution from a separate identifiable source (different lot number or vendor from that of calibration standards) immediately following the calibration. The result of the ICV solution must be within ±5% of the true value for NPDES compliance monitoring samples and ±10% for all other samples.

Corrective Action - If the calibration cannot be verified within the specified limits, re-analyze the ICV solution. If the results of the second analysis of the IPC/ICV solution are not within the acceptance limits for both types of samples (NPDES compliance monitoring samples and other samples), the analysis must be discontinued, the cause determined and the instrument re-calibrated. If the results of the second analysis of the IPC/ICV solution are not within the acceptance limits of NPDES requirements but within the acceptance limits for other program samples, a case narrative must be issued for samples that are non-compliant.

14.3 Continuing Calibration Verification (CCV)

Acceptance Criteria - Analyze the CCV solution, from the same source as that used for the ICV, after a maximum of ten samples and at the end of the sample run. The results of each CCV solution must be within ±10% of the true value for NPDES compliance monitoring samples and ±20% for all other samples.

Corrective Action - If the calibration cannot be verified within the specified limits, re-analyze the CCV solution. If the results of the second analysis of the CCV solution are not within the acceptance limits, the analysis must be discontinued, the cause determined and the instrument re-calibrated. All samples following the last acceptable CCV solution must be reanalyzed.

14.4 Initial Calibration Blank/Continuing Calibration Blank (ICB/CCB)

Acceptance Criteria - Analyze the calibration blank immediately following each calibration and after every CCV. All ICB/CCBs results must be < the |Reporting Limit.| for each element of interest..

Corrective Action - If the result of the ICB/CCB is > |Reporting Limit|, the analysis should be discontinued, the problem identified, and the ICB/CCB reanalyzed. If the ICB/CCB results remain > |Reporting Limit|, the instrument must be recalibrated.

14.5 Preparatory Blank (PB)/Laboratory Reagent Blank(LRB)

Acceptance Criteria - Analyze one PB/LRB per 20 samples or less per matrix. The PB/LRB results must be < the |Reporting Limit|.

Corrective Action - If the result of the PB/LRB is > |Reporting Limit|, then all associated samples with a concentration of ≤10x the amount found in the PB/LRB should be reprepared and reanalyzed. If the samples cannot be reprepared, then all affected sample results must be either: qualified accordingly, or the Reporting Limit is raised to the amount found in the sample. Check with the team leader/section chief to determine which option should be used.

Sample results ≥10x the amount found in the PB/LRB are not considered to be affected by the blank contamination or drift, so no corrective action is needed.

14.6 Laboratory Fortified Blank (LFB)/Laboratory Control Samples (LCS)

14.6.1 Aqueous LFB/LCS

Acceptance Criteria - Analyze two aqueous LFB/LCS samples with each batch of aqueous samples of 20 or less. Calculate accuracy as percent recovery using the following equation:

$$\%\ Recovery = \frac{Average\ of\ 2\ LFB/LCS's}{s}\ X\ 100$$

where: LFB/LCS = laboratory fortified blank/laboratory control sample result
s = concentration of analytes added to fortify the LFB/LCS solution

The %Recovery of the aqueous LFB/LCS must be within ±15% of the true value for NPDES wastewater compliance monitoring samples and within ±20% of the true value for all other environmental samples. The RPD of two LCSs should be <20%.

Corrective Action - If the %Recovery or %RPD results are outside the required control limits, the affected samples should be reprepared and reanalyzed. If the samples cannot be reprepared, then all affected sample results must be qualified accordingly.

14.6.2 Solid LCS

Acceptance Criteria - Analyze two solid LCS samples with each batch of solid samples of 20 or less. Calculate accuracy as percent recovery using the following equation:

$$\%\ Recovery = \frac{Average\ of\ 2\ LCS's, (mg/Kg)}{True\ Value, (mg/Kg)}\ X\ 100$$

The %Recovery of the solid LCS must be within ±25% of the true value or within the limits established by the vendor. The relative percent difference (RPD) of the duplicates should not exceed 25% for solid samples.

Corrective Action - If the %Recovery or %RPD results are outside the required control limits, the affected samples should be reprepared and reanalyzed. If the samples cannot be reprepared, then all affected sample results must be qualified accordingly.

14.7 Laboratory Fortified Matrix (LFM)/Matrix Spike(MS) Recovery

Acceptance Criteria - One Laboratory Fortified Matrix (LFM)/Matrix Spike (MS) is prepared for each matrix per project with at least one MS per batch of 10 or fewer NPDES samples or one MS per batch of 20 or fewer samples for other programs. The LFM/MS aliquot must be a duplicate of the aliquot used for sample analysis. When possible, the concentration should be the same as that added to the aqueous LFB/LCS, but should not exceed the midpoint concentration of the calibration curve. Calculate the percent recovery, corrected for background concentration measured in the unfortified sample aliquot, and compare these values to the control limits to the designated matrices' recovery ranges : ±20% for aqueous samples; ±25%

for solid samples (soils, sediment, and NAPL); and ±50% for sludge and biological tissue samples. Percent recovery is calculated using the following equation:

$$R = \frac{C_s - C}{s} X\ 100$$

where:
R = percent recovery,
Cs = fortified sample concentration,
C = sample background concentration, and
s = conc. equivalent of metal added to sample.

Corrective Action - If %Recovery of the MS is outside the required control limits, and the laboratory performance is shown to be in control, the recovery problem encountered is judged to be matrix related, not system related. The native sample result of the sample used to produce the MS must be qualified accordingly.

Note: The % recovery of the MS is not evaluated if the result of the unfortified sample concentration is ≥1.0x the level used to fortify the sample.

14.8 Serial Dilution Test

Acceptance Criteria - Analyze a 20% dilution of the MS sample(s). The serial diluted sample result(s), adjusted for the dilution, should agree with the MS result(s) to within 10% RPD.

Corrective Action - If the %RPD is outside the required control limits, and the laboratory performance is shown to be in control, the precision problem encountered is judged to be matrix related, not system related, and the sample should be qualified accordingly.

Calculation:

$$\% \text{ Difference} = \frac{|I - S|}{I} X\ 100$$

Where, I = Initial MS Result (Instrument reading)
S = Serial Dilution Result (Instrument reading x5)

14.9 Low-Level Checks - (RL and 2 RL)

Acceptance Criteria - Analyze the RL and 2 RL standards, immediately following the ICV and ICB and at the end of the run prior to the final IEC analysis. The %Recovery of the RL and 2 RL must be within ±30% of the true value for all analytes of interest.

Corrective Action –

If the RL cannot be verified within the specified limits of ± 30% for any analytes of interest, re-analyze the RL solution immediately for those analytes of concern only. If the results of the re-analysis for those analytes fall within the control limits, no further corrective action is required.

If the results of the RL re-analysis for those analytes do not fall within the control limits or if the RL standard cannot be reanalyzed, but the 2RL is within limits, then the Reporting Limit is raised to the 2 RL level as long as the sample project requirements allow. For projects where the 2RL levels are not acceptable the analysis must be discontinued; the cause determined, the instrument re-calibrated and samples re-analyzed. If the samples cannot be reanalyzed, then all affected sample results must be qualified accordingly.

If the 2RL cannot be verified within the specified limits of ± 30% for any analytes of interest, re-analyze the 2RL solution immediately for those analytes of concern only. If the results of the re-analysis for those analytes fall within the control limits, no further corrective action is required.

If the results of the 2RL re-analysis for those analytes do not fall within the control limits or if the 2RL standard cannot be reanalyzed, then the Reporting Limit may be raised to the next check standard (ICV/CCV or LCS), as long as the sample project requirements allow.

For projects where the 2RL levels are not acceptable, the analysis should be discontinued; the cause determined, the instrument re-calibrated and samples re-analyzed. If the samples cannot be reanalyzed, then all affected sample results must be qualified accordingly.

For samples with results > the next check standard (ICV/CCV or LCS), no further actions is necessary.

If the RL is acceptable, but the 2RL is unacceptable, samples with values >RL but < the next check standard (ICV/CCV or LCS), must be re-analyzed or qualified accordingly.

14.10 Spectral Interference Check Inter-Element Correction (IEC) Solution

Acceptance Criteria - All metal sample results, except for Al, Fe, Ca, Mg, and Na, should be below the established Reporting Limits listed in Table 2.

A corrective action is not required if one of the following conditions are met:

1. If the metal that is "affected" by the interferent is not required for the project in question;
2. If the concentration of the metal that is "affected" by the interferent is < the |Reporting Limit|;
3. If the concentration of the metal causing the interference in the "affected" environmental sample is at a trace level, i.e., <10,000 ug/L (the level used in our mixed calibration high

standard)

14.11 Triplicate Integrations

Acceptance Criteria - Each analysis consists of three separate integrations or readings. This includes the calibration standards, quality control samples and all associated environmental samples. The average of the three measurements is used for reporting results. The RSD must be ≤20% for all results that are ≥ the reporting limit.

Corrective Action - If the RSD for a calibration standard, quality control sample and environmental sample is outside the control limits, the analysis must be repeated. If the RSD is still outside the control limits, the analysis must be discontinued and repeated after correcting the problem. If the RSD for samples is still outside the control limits and the laboratory performance, i.e. CCV, is shown to be in control, the RSD problem encountered is judged to be matrix related, not system related, and the sample are qualified accordingly.

## 15. Reporting and Validation

15.1 Reporting Limits - The reporting limits are calculated based on MDL studies for each of the instruments and have been set wherever possible at from ½ CRQL levels, to the CRQL level, maximum. The reporting limits are matrix and dilution dependent. All results are reported to 2 significant figures.

15.2 Sample Data Package

The sample data package should include but not be limited to the following:

1. ICAP-AES QA/QC Checklist with all relevant information entered;
2. Copies of Log Book entries of Analysis Run Log; and Sample Digestion Log
3. Calibration Report;
4. Summary Analysis Form;
5. QC Summary Forms; and
6. Instrument generated Sample raw data
7. Project Data Cross Reference Form

15.3 Laboratory Information Management System (LIMS) - The analyst enters the data on the LIMS under the appropriate analytical codes.

15.4 Data Validation - The data package is given to the reviewer. The review is done by a peer who was not involved in the analysis. Upon completion of the review, including validation of all the appropriate codes in the LIMS for the particular project(s), the data reviewer will sign and date the QA/QC Checklist.

15.5 Data Records - All project records associated with the data package are filed under one designated project file. All other projects associated with the data package are referenced to this designated project file via a "cross reference form". The "cross reference form" is placed in each of the project files that were associated with the data package.

The data package is placed in the bin identified for the designated project file. The records for this designated project file are filed in our locked record cabinets once all data from the project has been reviewed by the appropriate staff.

## 16. Pollution Prevention

16.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

16.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

16.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult Less is Better: Laboratory Chemical and Management for Waste Reduction, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202)872-4477.

## 17. Waste Management

The USEPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process waste should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the Region 2 SOP G-6, "Disposal of Samples and Hazardous Wastes".

## 18. References

- U. S. Environmental Protection Agency. *"Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry,"* Method 200.7, Revision 4.4, May 1994.
- U. S. Environmental Protection Agency, Region 2, SOP G-6 "*Disposal of Samples and Hazardous Wastes."*
- U. S. Environmental Protection Agency, Region 2, SOP G-8 "*Laboratory Policy for the Determination of Method Detection Limits (MDLs)."*
- . S. Environmental Protection Agency, Region 2, SOP G-15 *"Laboratory Definitions and Data Qualifiers."*
- U. S. Environmental Protection Agency, Region 2, SOP G-23 *"Percent Dry Solids."*
- U. S. Environmental Protection Agency, Region 2, SOP C-116 *"Preparation of Aqueous, TCLP Extracts, Soil/Sediment/Sludge, Waste Oil/Organic Solvents, and Biological Tissue Matrices by Block Digestion."*
- Method 2340 B *"Hardness by Calculation"*_Standard Methods for the Examination of Water and Wastewater, 20th Edition-1998.
- U. S. Environmental Protection Agency, Solid Waste 846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 6010C *"Inductively Coupled Plasma - Atomic Emission Spectrometry"* Laboratory Manual, Revision 3, November 2000.

**Table 1. Standard Solutions Preparation:**

| Standard/Solution Name | Concentration Required |
|---|---|
| Calibration Blank/Rinse Blank/ICB/CCB | Reagent grade water, acidified to 2% HNO3 and 5% HCl |
| High Standard - IRIS | 10,000 ppb for Al, Ca, Fe, Mg, K, Na and Si<br>1000 ppb for Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Sr, Ti, Tl, V, and Zn |
| Mid Standard - IRIS | 5,000 ppb for Al, Ca, Fe, Mg, K, Na and Si<br>500 ppb for Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Sr, Ti, Tl, V, and Zn |
| ICV/CCV IRIS | 200 ppb for Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Sr, Ti, Tl, V, and Zn<br>5,000 ppb for Al, Ca, Fe, Mg, K, Na and Si |
| RL | All elements at Reporting Limit levels |
| 2 RL | All elements at 2X Reporting Limit levels. |
| IEC Solution | 300,000 ppb Al, Ca, Fe, Mg and Na |
| High Standard – iCAP 6300 | 10,000 ppb for Al, Ca, Fe, Mg, K, Na ,Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Ti, Tl, V, and Zn<br>1,000 ppb for Ag |
| Mid Standard – iCAP 6300 | 5,000 ppb for Al, Ca, Fe, Mg, K, Na ,Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Ti, Tl, V, and Zn<br>500 ppb for Ag |
| ICV/CCV iCAP 6300 | 100 ppb for Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Ti, Tl, V, and Zn<br>2,,000 ppb for Al, Ca, Fe, Mg, K, and Na |
| Internal Standard IRIS | 10 ppm Y |
| Internal Standard iCAP 6300 | 3,000 ppm Cs, 10 ppm Y |

**Table 2. IRIS - Reporting Limits - Aqueous and Soil/Sediment - LDR**

| Element/ Wavelength (nm) | IIDL µg/L | MDL µg/L | Reporting Limit µg/L - Aqueous | Reporting Limit mg/Kg Soil | LDR ppm |
|---|---|---|---|---|---|
| Ag 328.0 A | 0.970 | 3.49 | 5 | 0.5 | 2 |
| Al 396.1 R | 27.58 | 99.3 | 100 | 10 | 800 |
| As 189.0 A | 1.887 | 6.79 | 8 | 0.8 | 25 |
| B 208.9 A | 0.424 | 1.53 | 10 | 1 | 100 |
| Ba 455.4 R | 1.030 | 3.71 | 100 | 10 | 100 |
| Be 313.1 R | 0.553 | 1.92 | 3 | 0.3 | 10 |
| Ca 317.9 R | 20.69 | 74.5 | 500 | 50 | 500 |
| Cd 214.4 A | 0.08 | 0.322 | 3 | 0.3 | 12.5 |
| Co 228.6 A | 0.233 | 0.840 | 20 | 2 | 40 |
| Cr 205.5 A | 0.166 | 0.598 | 5 | 0.5 | 40 |
| Cu 324.7 A | 1.042 | 3.75 | 10 | 1 | 40 |
| Fe 259.9 A | 0.787 | 2.83 | 50 | 5 | 0-25 |
| Fe 259.9 R | 21.31 | 76.7 | 100 | 10 | 0-300 |
| K 766.4 R | 133.9 | 482 | 500 | 50 | 400 |
| Mg 279.0 R | 112.0 | 403.2 | 500 | 50 | 0-1000 |
| Mg 285.2 R | 11.33 | 40.78 | 500 | 50 | 0-100 |
| Mn 302.0 A | 0.123 | 0.442 | 5 | 0.5 | 7 |
| Mo 202.0 A | 0.160 | 0.577 | 10 | 1 | 8 |
| Na 589.5 R | 177.2 | 638 | 1000 | 100 | 1000 |
| Ni 231.6 A | 0.488 | 1.76 | 20 | 2 | 75 |
| Pb 220.3 A | 1.263 | 4.55 | 8 | 0.8 | 200 |
| Sb 206.8 A | 1.595 | 5.74 | 20 | 2 | 150 |
| Se 196.0 A | 2.593 | 9.334 | 20 | 2 | 50 |
| Si 251.6 R | 50.49 | 182 | 500 | 50 | 400 |
| Si 251.6 A | 1.509 | 5.43 | 10 | 0.1 | 5 |
| Sn 189.9 A | 1.076 | 3.87 | 10 | 1 | 15 |
| Sr 407.7 R | 0.405 | 1.46 | 10 | 1 | 30 |
| Ti 334.9 A | 0.326 | 1.17 | 10 | 1 | 15 |
| Tl 190.8 A | 2.008 | 7.23 | 20 | 2 | 200 |
| V 272.4 A | 0.897 | 3.23 | 20 | 2 | 50 |
| Zn 206.2 A | 0.477 | 1.72 | 20 | 2 | 20 |

Notes

1) Seven blanks were used to determine the IDLs for the IRIS
2) The IDLs for all elements, were obtained by multiplying the standard deviation of the seven analyses by 3.14
3) The MDLs for all elements were obtained by multiplying the IDL by 3.6.
4) The Reporting Limits were set wherever possible at ½CRQL levels, maximum. Pb is 0.8X CRQL level.
5) A: Axial View, R: Radial View

**Table 3 Reporting Limits - Aqueous and Soil/Sediment for iCAP 6300 – LDR**

| Element/ Wavelength (nm) | MDL µg g/L | Reporting Limit µg /L, Aqueous | Reporting Limit mg/Kg Soil | LDR ppm |
|---|---|---|---|---|
| Ag 328.0 A | 1.33 | 5 | 0.5 | 5 |
| Al 308.2 A | 26.5 | 100 | 10 | 350 |
| As 189.0 A | 4.80 | 8 | 0.8 | 100 |
| B 208.9A | 2.46 | 10 | 1.0 | 100 |
| Ba 455.4 R | 27.6 | 100 | 10 | 90 |
| Be 313.1 R | 1.44 | 3 | 0.3 | 75 |
| Ca 317.9 R | 133 | 500 | 50 | 500 |
| Cd 226.5 A | 1.46 | 3 | 0.3 | 100 |
| Co 228.6 A | 5.44 | 20 | 2 | 100 |
| Cr 267.7 A | 2.90 | 5 | 0.5 | 100 |
| Cu 324.7 A | 5.03 | 10 | 1.0 | 100 |
| Fe 259.9 A | 14.2 | 50 | 5 | 100 |
| Fe 259.9 R | 13.7 | 50 | 5 | 100 |
| K 766.4 R | 154 | 500 | 50 | 600 |
| Mg 279.0 R | 139 | 500 | 50 | 1000 |
| Mn 257.6 A | 3.04 | 5 | .5 | 50 |
| Mo 202.0 A | 2.70 | 10 | 1 | 100 |
| Na 589.5 R | 274 | 1000 | 100 | 800 |
| Ni 231.6 A | 5.43 | 20 | 2 | 95 |
| Pb 220.3 A | 2.39 | 8 | 0.8 | 80 |
| Sb 206.8 A | 11.2 | 20 | 2 | 100 |
| Se 196.0 A | 11.2 | 20 | 2 | 95 |
| Si 288.1A | 24.9 | 500 | N/A | 500 |
| Si 288.1R | 34.4 | 500 | N/A | 550 |
| Sn 189.9A | 1.37 | 10 | 1.0 | 100 |
| Sr 346.4 | 1.48 | 10 | 1.0 | 100 |
| Ti 337.2 A | 2.91 | 10 | 1.0 | 95 |
| Tl 337.2 A | 7.58 | 20 | 2 | 100 |
| V 292.4 A | 5.62 | 20 | 2 | 100 |
| Zn 206.2 A | 5.71 | 20 | 2 | 100 |

Notes

1) IDL values were not determined for the iCAP 6300
2) A: Axial View, R: Radial View

# REQUEST FOR SOP CHANGE

Initiator Name: Renee Lettieri Date of Initiation: 4/30/2010

Dept: Metals

SOP Title:
**DETERMINATION OF METALS IN AQUEOUS, TCLP EXTRACTS, SOIL/SEDIMENT, SLUDGE, AND BIOLOGICAL TISSUE SAMPLES BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY**

SOP #: C-109 Revision #: 3.0

Please Check One

MINOR REVISION X MAJOR REVISION

**CHANGE(S) (Use attachment if necessary):**
Delete

Table 2 - IRIS - Reporting Limits - Aqueous and Soil/Sediment - LDR
And

Table 3 Reporting Limits - Aqueous and Soil/Sediment for iCAP 6300 – LDR

---

**REASON(S) FOR CHANGE(S):**

**MDL and LDR values no longer included as part of SOP**

| APPROVAL: | NAME: | Signature/Date |
|---|---|---|
| EPA Section Chief/Team Leader | JOHN BOURBON | 5-27-10 |
| ESAT Analytical Supervisor/QAO | | |
| EPA Task Order Project Officer | | |

Effective Date: 05/27/10

Sumy Cherukara
EPA QAO

# REQUEST FOR SOP CHANGE

Initiator Name: Renee Lettieri Date of Initiation: 10/21/09

Dept: Metals

SOP Title: Metals By ICP-AES

SOP #: C109 Revision #: 3.0

Please Check One

MINOR REVISION X MAJOR REVISION ☐

**CHANGE(S) (Use attachment if necessary):**

Page 24 of 25 – Table 2 – Reporting Limits – Aq-Soil/Sediment - LDR

Correct Typo

Change wavelength for Mn from 302.0 A to 257.6 A.

---

**REASON(S) FOR CHANGE(S):** Typographical Error

| **APPROVAL:** | **NAME:** | **Signature/Date** |
|---|---|---|
| EPA Section Chief/Team Leader | Philip Cocuzza | Philip Cocuzza 10/21/09 |
| ESAT Analytical Supervisor/QAO | | |
| EPA Task Order Project Officer | | |

Effective Date: 11/3/09

Sumy Cherukara
EPA QAO

# REQUEST FOR SOP CHANGE

**Initiator Name:** Renee Lettieri **Date of Initiation:** July 15, 2010

**Dept :** Metals **SOP #** C-109Determination of Metals by ICP-AES

**SOP Revision #:** 3.0

Please Check One MINOR REVISION [X] MAJOR REVISION [ ]

**CHANGE(S) (Use attachment if necessary):**

Change Section 7.2 to read:

Purchased standard solutions may be used until the expiration date given by the manufacturer or until one year after being opened if no date is given. Prepared solutions may be used for one year from date of preparation unless a shorter time is specified. Refer to Table 1 for standard solutions preparation summary. Solutions are prepared using 2% $HNO_3$ and 5% HCl.

**REASON(S) FOR CHANGE(S):**
An expiration date is listed for purchased and prepared standards and reagents.
The reference to UL ISO 9001 certification was removed.

| APPROVAL | NAME: | Signature/Date |
|---|---|---|
| EPA Section Chief/Team Leader | JOHN BOURBON | 7-16-10 |
| ESAT Analytical Supervisor/QAO | | |
| EPA Task Order Project Officer | | |
| Effective Date: 7/30/10 | Sumy Cherukara EPA QAO | 7/30/10 |

USEPA Region 2
Division of Environmental Science & Assessment
Laboratory Branch

EPA

# STANDARD OPERATING PROCEDURE

# DETERMINATION OF TRACE ELEMENTS IN AQUEOUS, SOIL/SEDIMENT, SLUDGE, WASTE OIL/ORGANIC SOLVENTS, AND BIOLOGICAL TISSUE SAMPLES BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY

*Signature and Title*

| | | |
|---|---|---|
| Prepared by: | Renee Lettieri | 5-10-10 |
| | Renee Lettieri, Chemist, OICS | Date |
| Peer Reviewed by: | | 5/10/10 |
| | Ness Tirol, Chemist, OICS | Date |
| QA Reviewed by: | | 05/10/10 |
| | Sumy P. Chelukara, Quality Assurance Officer | Date |
| Approved by: | J. R. Bourbon | 5-10-10 |
| | John R. Bourbon, Chief, OICS | Date |
| Approved by: | J. R. Bourbon | 5-10-10 |
| | John R. Bourbon, Acting Chief, Laboratory Branch | Date |

*Annual Review*

| | | |
|---|---|---|
| Reviewed by: | | |
| | Signature | Date |
| Reviewed by: | | |
| | Signature | Date |

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION 2
DIVISION OF ENVIRONMENTAL SCIENCE AND ASSESSMENT
LABORATORY BRANCH

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## TABLE OF CONTENTS

# DETERMINATION OF TRACE METALS IN AQUEOUS, SOIL/SEDIMENT, WASTE OIL/ORGANIC SOLVENTS, AND BIOLOGICAL TISSUE MATRICES BY BY INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY

## 1. Scope and Application

1.1 This SOP is applicable to the preparation and analysis of environmental samples, e.g., aqueous, soil/sediment, biological tissue, and waste oil/organic solvents, for the determination of the following metals:

Ag, Al, As, Ba, Be, Cd, Co, Cu, Cr, Mn, Mo, Ni, Pb, Se, Sb, Tl, Th, U, V, and Zn. Refer to Table 1 for recommended elemental isotopes for this method.

NOTE - This SOP is not applicable to the preparation and analysis of drinking water compliance monitoring samples. The procedure for the preparation and analysis of drinking water compliance monitoring samples using ICP-MS is detailed in Laboratory SOP DW-8.

1.2 The range of the method for most of the elements is 0.25 to500.0 µg/L. The lowest standard concentration of the curve generated is used as the basis for the reporting limit for all types of sample matrices.

1.3 This SOP is based on EPA Method 200.8 Revision 5.4.

1.4 All analysts must satisfactorily perform an initial demonstration of capability (DOC) by meeting the method performance criteria in Sec. 13.1 prior to performing sample analysis using this SOP.

## 2. Summary of SOP

2.1 An aliquot of a well mixed, homogenous aqueous or soil sample is accurately weighed or measured for sample processing. For total recoverable analysis of a solid or an aqueous sample containing undissolved material, elements are first solubilized by gentle refluxing with $HNO_3$ and HCl. After cooling, the sample is made up to volume, mixed and filtered (if necessary) prior to analysis. For the determination of dissolved elements in a filtered aqueous sample, where the turbidity is <1 NTU, the sample, acidified with $HNO_3$, may be analyzed directly

2.2 The method describes the multi-element determination of trace element by ICP-MS. Sample material in solution is introduced by pneumatic nebulization into a radio-frequency plasma where energy transfer processes cause desolvation, atomization and ionization. The ions are extracted from the plasma through a differentially pumped

vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer having a minimum resolution capability of 1 amu peak width at 5% peak height. The ions transmitted through the quadrupole are detected by an electron multiplier and the ion information processed by a data handling system. Interferences relating to the technique must be recognized and corrected for. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents or sample matrix. Instrumental drift as well as suppressions or enhancements of instrument response caused by the sample matrix must be corrected for by the use of internal standards.

## 3. Definitions

See SOP G-15 for definitions.

## 4. Interferences

Several interferences sources may cause inaccuracies in the determination of trace elements by ICP-MS. These are:

4.1 Isobaric elemental interferences - Are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for the use with this method, only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process should be included with the report of the data. It should be noted that such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios should be established prior to the application of any corrections.

4.2 Abundance sensitivity - Is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

4.3 Isobaric polyatomic ion interferences - Are caused by ions consisting of more than

one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma of interface system from support gases or sample components. Most of the common interferences have been identified in Table 3 together with the method elements affected. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Equations for the correction of data should be established at the time of the analytical run sequence as the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions. In particular, the common 82Kr interference that affects the determination of both arsenic and selenium can be greatly reduced with the use of high purity krypton free argon.

Analysis using Collision/Reaction technology is useful for the removal of most isobaric polyatomic ion interferences and is permitted for waste water and non regulated samples but is not yet approved for drinking water.

4.4 Physical interferences - Are associated with the physical processes which govern the transport of sample into the plasma, sample conversion process in the plasma, and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. Dissolved solids levels not exceeding 0.2% (w/v) have been recommended to reduce such effects. Internal standards ideally should have similar analytical behavior to the elements being determined.

4.5 Memory interferences - Result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones and from the sample buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce element signals to within a factor of ten of the method detection limit, should be noted. Memory interferences may also be assessed within an analytical run by using a minimum of

three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alerted to the possibility of a memory effect, and should examine the element concentration in the previous sample to identify if this was high. If memory interference is suspected, the sample should be reanalyzed after a long rinse period.

## 5. Safety

The toxicity and carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices, e.g. wear proper protective equipment, safety glasses, gloves, lab coat and working inside hoods whenever possible. Refer to Edison Facility Safety Manual Region II (available on the Region II Intranet), Part 2 – Laboratory Safety and Appendices 13/13A - Chemical Hygiene Plan for specific guidelines.

## Apparatus and Materials

6.1 Agilent 7700X ICP-MS - capable of scanning the mass range 2 – 260 amu and equipped with:

6.1.1 AMFC – Active Mass Flow Control - electronically controls Argon and Cell Gas Flows

6.1.2 Variable-speed peristaltic pump - for solution delivery to the nebulizer

6.1.3 Nebulizer, Peltier cooled spray chamber and Torch

6.1.4 RF generator -, 27.12MHz, 500 to 1600 W - to generate the plasma

6.1.5 Internal Vacuum system – Single split flow turbo pump backed by single rotary pump

6.1.6 Interface and Ion Optics system

6.1.7 Octopole reaction system for operation in optional Collision/Reaction modes

6.1.8 Quadrupole Mass Spectrometer

6.1.9 Electron Multiplier Dual Mode Detector (digital/analogue)

6.1.10 Mass Hunter Software with Intelligent Sequencing

6.1.11 ISIS - Integrated Sample Introduction System (optional)

6.1.12 HMI – High Matrix Interface for optional use with high TDS (Total Dissolved Solids) samples

6.2 Heat exchanger – PolyScience Model 3370 Air Cooled Recirculator

6.3 External Rough Pump – Edwards Model E2M18

6.4 Computer, monitor and printer

6.5 Autosampler – Cetac for Agilent ASX-520 Autosampler with anti-contamination enclosure

6.6 High purity grade liquid argon (99.99%) and optional Helium (99.999%) and Hydrogen (99.999%) with gas purification assembly

6.7 Labware - See Section 6.10 of EPA Method 200.8 Rev. 5.4

Important Note: - Chromic acid must not be used for cleaning glassware.

6.7.1 Assorted Glassware - Volumetric flasks, graduated cylinders, funnels and centrifuge tubes (glass and/or metal free plastic)

6.7.2 Assorted calibrated battery operated pipettes

6.7.3 Storage bottles: Polyethylene (low and high density), Polypropylene, Fluoropolymers (Teflon, PTFE, PFA and FEP) and Pyrex glass are commonly used to store Standards and prepared Reagents

6.7.4 Test tubes for Autosampler, polypropylene, 15 mL, (17x100 mm)

## 7. Reagents and Solutions

7.1 Reagents

All acids used for this method must be of ultra high purity grade suitable for ultra trace metals analysis. $HNO_3$ is preferred for ICP-MS in order to minimize polyatomic ion interferences. Several polyatomic ion interferences result when HCl is used (see Table 3), however, it should be noted that HCl is required to maintain stability in solutions containing Sb and Ag. When HCl is used, corrections for the chloride polyatomic ion interferences must be applied to all affected data. Use of the Helium collision cell effectively eliminates chloride interference.

7.1.1 Nitric acid, concentrated - $HNO_3$ (GFS $HNO_3$, Double Distilled, or equivalent)

7.1.2 Hydrochloric acid, concentrated - HCl (GFS HCl, 30-35%, Reagent ACS, or equivalent)

7.1.3 Reagent grade water – ASTM Type I Water

7.2 Solutions:

Purchased Standard Solutions may be used until the expiration date given by the manufacturer or until one year after being opened if no expiration date is given. Diluted standards prepared in the lab expire six months after preparation.

Standard solutions equivalent to those listed below with dilutions adjusted as necessary may be substituted for those listed below.

7.2.1 Calibration Stock Standard Solutions - Spex Instrument Calibration Standard Stock Solution, CL-CAL-1, (20ppm Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn) in $HNO_3$ matrix or equivalent.

7.2.2 Aluminum Stock Solution – Absolute, 10,000ppm or equivalent.

7.2.2.1 Aluminum Intermediate Solution, 250 ppm – dilute 1.25 mL Aluminum Stock Solution, 10,000 ppm to 50 mL with 2% $HNO_3$.

7.2.3 Calibration Standard – Dilute 12.5 mL Calibration Stock Standard (20 ppm) and 9 mL Aluminum Intermediate solution (250 ppm) to 500 mL with 2% $HNO_3$. This solution will contain 500 ppb Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn and 5000 ppm of Al.

7.2.4 Working Calibration Standards are prepared by diluting the 500 ppb Intermediate Calibration Stand according to the following table with 2% $HNO_3$. Standards may be added or removed depending on analytical needs and/or instrument operation.

| Solution Name | Intermediate. Cal. Std - 500 ppb | Final Volume 2% $HNO_3$ | Concentration Metals | Concentration Al (ppb) |
|---|---|---|---|---|
| Cal std 0.25 ppb | 0.125 mL | 250 mL | 0.25 ppb | N/A |
| Cal Std 0.5 ppb | 0.25 ml | 250 mL | 0.5 ppb | N/A |
| Cal Std 1 ppb | 0.5 mL | 250 mL | 1 ppb | N/A |
| Cal Std 2 ppb | 1.0 mL | 250 mL | 2 ppb | 20 ppb |
| Cal Std 5 ppb | 2.5 mL | 250 mL | 5 ppb | 50 ppb |
| Cal Std 20 ppb | 10 mL | 250 mL | 20 ppb | 200 ppb |
| Cal Std 50 ppb | 25 mL | 250 mL | 50 ppb | 500 ppb |
| Cal Std 100 ppb | 50 mL | 250 mL | 100 ppb | 1000 ppb |

7.2.4.1 Working Calibration Standard, 250 ppb Metals, 2500 ppb Aluminum -Dilute 3.125 mL Calibration Stock Standard (20 ppm) and 2.25 mL Aluminum Intermediate Solution (250 ppm) to 250 mL with 2% $HNO_3$. This solution will contain 250 ppb Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn and 2500 ppm of Al.

7.2.4.2 Working Calibration Standard, 500 ppb Metals, 5000 ppb aluminum – The Intermediate Calibration Standard can also be used as a Working calibration Standard. This solution contains 500 ppb Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn and 5000 ppm of Al.

7.2.3 Internal Standard Stock Solution – Spex Multi-Element Internal Standard (Cat. #CLISS-1) or equivalent. This solution contains 10 mg/L Lithium6, Scandium, Yttrium, Indium, Terbium, Holmium and Bismuth

7.2.3.1 Internal Standard Solution, 1000 ppb – Dilute 50 mL Internal Standard Stock Solution (10 ppm) to 500 mL with 2% $HNO_3$. This solution is added in line to all analyzed solutions (blanks, calibration standards and samples) via the peristaltic pump and mixing "T". (Alternatively, this solution may be added in appropriate amounts, individually to blanks, calibration standards and samples.)

7.2.4 Blanks – Four types of blanks are required for the analysis. The calibration blank is used in establishing the analytical curve; the laboratory reagent blank or prep blank is used to assess possible contamination from the sample preparation procedure and to assess spectral background; the instrument performance check blank (ICB/CCB) is run after the instrument performance check (ICV/CCV) to assess carryover; and the rinse blank used to flush the instrument uptake system

and nebulizer between standards, check solutions, and samples to reduce memory interferences.

7.2.4.1 The Calibration Blank, Instrument Check Blank (ICB/CCB) and Rinse Blank – are prepared by acidifying reagent grade water to the concentration of 2% v/v $HNO_3$.

7.2.4.2 Laboratory Reagent Blank (LRB)/Prep Blank (PB) - must contain all the reagents in the same volumes as used in preparing the samples. The LRB/PB must be carried through the same preparation scheme as the samples including digestion, if applicable.

7.2.5 Tuning Solution, Stock – Agilent ICP-MS Stock Tuning Solution, 10mg/L: Li, Y, Ce, Tl, Co or equivalent.

7.2.5.1 Tuning Solution for no gas mode, 1 ppb – Dilute 0.1 mL Stock Tuning Solution to 1 Liter with 2% $HNO_3$.

7.2.5.1 Tuning Solution for He mode, 1 ppb – Dilute 0.1 mL Stock tuning Solution and 10 mL concentrated HCL to 1 Liter with 2% $HNO_3$.

7.2.6 P/A Tuning Solution – P/A factor tuning is used to achieve linear functionality across the detector's pulse and analogue modes.

7.2.6.1 P/A Tuning Solution 1, Stock- Agilent Part # 5188-6524.

7.2.6.1.1 P/A Tuning Solution 1(1:75) – Dilute 3 mL P/A Stock Tuning Solution 1, to 225 mL with 2% $HNO_3$.

7.2.6.2 P/A Tuning Solution 2, Stock - Agilent Part # 5188-6524

7.2.6.2.1 P/A Tuning Solution 2 (1:75) – Dilute 3 mL P/A Stock Tuning Solution 2, to 225 mL with 2% $HNO_3$.

7.2.6.3 P/A Tuning Solution 1+2 – Combine equal volume of P/A Tuning solution 1 and 2.

7.2.7 Instrument Performance Check (ICV/CCV) – The instrument performance check is used to initially and periodically verify instrument performance during analysis. The instrument performance check stocks must be obtained from a source different from the calibration stock standard solutions (different vendor or lot #) and prepared in the same acid mixture as the calibration standards.

The concentration of the ICV/CCV solution may be varied to accommodate special requirements for non routine projects. Acceptance criteria will remain

the same

ICP-MS Calibration Standard 1 Stock, Accutrace, 10 mg/L: Al, Sb, As, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, V, Zn.

ICP-MS Calibration Standard 2, Stock Accutrace, 10 mg/L: Ba, Ag.

7.2.7.1 ICV/CCV 10 ppb – Dilute 0.25 mL each Accutrace ICP-MS Calibration Standards 1 and 2 to 250 mL with 2% $HNO_3$.

7.2.7.2 ICV/CCV 50 ppb – Dilute 1.25 mL each Accutrace ICP-MS Calibration Standards 1 and 2 to 250 mL with 2% $HNO_3$. This standard is used for verification for Aluminum.

Or alternatively:

Instrument Check Standard 1, Spex (CL-ICS-1), 10mg/L: Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Sb, Se, Tl, V, Zn. (contains no Mo).

7.2.7.3 ICV/CCV 10 ppb - Dilute 0.25 mL Instrument Check Standard 1, Spex (CL-ICS-1), 10mg/L to 250 mL with 2% $HNO_3$

7.2.7.4 ICV/CCV 50 ppb - Dilute 1.25 mL Instrument Check Standard 1, Spex (CL-ICS-1), 10mg/L to 250 mL with 2% $HNO_3$. This standard is used for verification for Aluminum.

7.2.8 ICP-MS Low Level LCS/Spike Solutions:

7.2.8.1 Trace Metals Stock - SPEX CertiPrep Custom Calibration Standard 1 (250 ppm of Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Sr, Ti, Tl, V, and Zn) or equivalent.

7.2.8.2 Trace Metals - ICP-MS Low Level LCS/Spike – Dilute 0.4 mL Calibration Standard 1 to 100 ml with 2% $HNO_3$. This solution contains 1000 ppb of the above listed trace Metals.

7.2.8.3 Minerals Stock - SPEX CertiPrep Custom Calibration Standard 2 - (250 ppm of Al, Ca, Fe, Mg, K, Na and Si) or equivalent.

7.2.8.4 Minerals - ICP-MS Low Level LCS/Spike – Dilute 4 mL Calibration Standard 2 to 100 mL using 2% $HNO_3$. This solution contains 10 ppm of the above listed Minerals.

7.2.8.5 LCS/Spike Values: 1 mL of each of these ICP-MS Solutions Low Level Solutions added to a 50 mL digestion tube will have a an LCS/Spike content

of 20 ppb Trace Metals and 200 ppb Aluminum.

For directly analyzed samples, pipet 0.2 mL Trace Metals ICP-MS Low Level LCS/Spike, 1000 ppb and/or 0.2 mL Minerals - ICP-MS Low Level LCS/Spike, 10 ppm into a 15 mL test tube. Dilute to 10 ml with sample. The sample will have a. Spike content of 20 ppb Trace Metals and/or 200 ppb Minerals.

The amount added for LCS/Spike should be the same as that added to the aqueous LFB/LCS. It can be varied depending on analytical needs, reporting requirements and/or instrument operation but should not exceed the midpoint of the standard curve.

The Minerals LCS/Spike need not be added for projects that do not request Aluminum.

The two solutions may be prepared separately as described above or may be prepared combined as a single solution.

## 8. Sample Collection, Preservation, Storage and Holding Time

8.1 Sample Collection - Samples must be collected in plastic or glass containers.

8.2 Preservation and Storage

8.2.1 Aqueous samples - are preserved to a pH <2 using concentrated $HNO_3$. The preservation is performed either a) in the field at the time of collection, or b) in the Laboratory upon receipt (within five days). If the samples are preserved in the Laboratory, they must be held for sixteen hours after acidification and then verified to a pH < 2 prior to sample processing. If the sample pH is verified to be pH > 2 after the sixteen hours, additional $HNO_3$ must be added and the sample held for an additional sixteen hours until verified to a pH < 2. The samples are stored at room temperature.

8.2.2 Soil/Sediment/Sludge samples - are preserved in a refrigerator at 4°C. Alternatively, the samples maybe stored at -20°C in a freezer.

8.2.3 Biological Tissue samples - are stored at -20°C in a freezer.

8.2.4 Waste Oil/Organic Solvents - do not require any preservation. The samples are stored at room temperature.

8.2.5 Drum Samples - no temperature requirement for these samples.

8.3 Holding time

8.3.1 Aqueous samples must be prepared and analyzed within six months of collection.

8.3.2 Soil/Sediment/Sludge samples must be digested and analyzed within six months of collection. If soil/sediment samples are stored at -20°C, the holding time is extended. The samples must be prepared within 12 months of collection and analyzed within 6 months of digestion.

8.3.3 Biological Tissue samples must be digested within 12 months of collection and analyzed within 6 months of digestion.

8.3.4 Waste Oil/Solvent samples - a holding time is not established for the digestion of these samples. Samples must be analyzed within 6 months of digestion.

8.3.5 Drum Samples do not have any established holding time. Samples must be analyzed within 6 months of digestion.

## 9. Sample Preparation

9.1 All Environmental samples, e.g., aqueous, soil/sediment, waste oil/organic solvent, and biological tissue, including NPDES wastewater compliance monitoring samples, are digested in a mixture of acids using the procedures described in SOP # C-116. "Digestion of Metals Aqueous, TCLP Extracts, Soil/Sediment, Sludge, Waste Oil/Organic Solvents, TCLP Extracts and Biological Tissue Matrices by DigiBloc".

9.2 For the determination of dissolved elements in a filtered aqueous sample, where sample turbidity is <1 NTU, the sample, acidified with $HNO_3$, is analyzed directly.

## 10. Instrument Operating Conditions

Before using this method, the following procedure is to be followed to optimize plasma conditions. The analyst should follow the Agilent 7700X instructions unless other conditions provide better performance.

10.1 Instrument Set-Up Up - Before lighting the plasma, check the following settings are in place:

10.1.1 Argon gas tank pressure to the instrument should be set at 500 to 700 kPa. Cell gases, if used, should be set at:,
Helium – 90 to 130 kPa,
Hydrogen – 20 to 60 kPa

10.1.2 The Exhaust Duct is on and is recommended to remain on at all times when Hydrogen is connected to the instrument and to prevent condensation from the outside.

10.1.3 The heat exchanger is turned on.

10.1.4 The Instrument is in standby mode.

10.1.5 The peristaltic tubings for the sample, drain and internal standard lines are not worn out and are connected properly. Loosen the peristaltic tubings when not in use.

10.2 Instrument Start-Up:

Open the ICP-MS Instrument control panel in the Mass Hunter Software and confirm that the instrument is in Standby Mode. Ignite the plasma by clicking the Ignite Plasma icon in the toolbar. Allow the instrument to warm up for 15 to 30 minutes.

10.3 Instrument Tuning – should be performed prior to each day's use. Tune first in no gas mode, then in Helium Mode and Hydrogen Mode, if being used.

10.3.1 No Gas Tune:

a) Open the Tune window and load the last used Tune file for no gas mode.

b) Acquisition parameters should remain at 7(Li), 89(Y), 205(Tl) and 156/140 (Cesium Oxide formation rate).

c) Send the autosampler probe to the tuning solution (1 ppb Li, Y, Tl, Ce, Co) and hit "Start" to aspirate the tuning solution. Observe the tuning sensitivity window to assure that the solution has reached the plasma.

With the integration time set at 0.1 second (default), sensitivities should read:

| | |
|---|---|
| 7 Li | > 3,000 counts/0.1 second |
| 89 Y | > 10,000 counts/0.1 second |
| 205 Tl | > 6,000 counts/0.1 second |

RSD's should be < 5%
Oxide Formation Ratio should be < 1.2% (0.7% to 1.0% best optimizes the sensitivity and noise level)

d) For manual tune, minor adjustments may be made to the Ion Lenses, Octopole and Quadrupole parameters to optimize sensitivity. The carrier gas may be adjusted to improve the oxide level. Any necessity for large adjustments will trigger the need for a troubleshooting session.

e) Save the Tune File as ngymmdd.u. This file needs to be loaded into the method before that day's analysis run.

f) To generate a Tune Report, click on File → Generate Report. Input comments, then click OK. A report showing Sensivity data, Resolution/Axis data and the current lens parameter settings will be printed. Tuning results are also recorded in the maintenance log.

g) The instrument may also be tuned by selecting the Autotune function.

10.3.2 Helium Mode Tune:

a) Aspirate the Helium Tune Solution. Observe the tuning sensitivity window to assure that the solution has reached the plasma.

b) Load the last used Tune file for Helium mode.

c) Acquisition parameters should remain at 59(Co), 78/59 (Ar2/Co), 75/59 (ArCl/Co) and 51/59 (ClO/Co).

d) Set the Helium Gas Flow at 4 ml/minute (range 3-5 ml/minute). Adjust the gas settings to achieve required interference reduction.

e) Adjust the lens settings: QP bias, Octopole Bias, Deflect, Cell exit, Cell entrance.

f) Save the tune file as Heymmdd.u. Print the Tune page.

10.3.3 Hydrogen Mode Tune

a) Aspirate the Tune Solution. Observe the tuning sensitivity window to assure that the solution has reached the plasma.

b) Load the last used Tune file for Hydrogen mode.

c) Acquisition parameters should remain at 59 (Co), 28/59, 28, and 78/59(Ar2/Co).

d) Set the Hydrogen Gas Flow at 4 ml/minute (range 3-5 ml/minute). Adjust the gas settings to achieve required interference reduction.

e) Adjust the lens settings: QP bias, Octopole Bias, Deflect, Cell exit, Cell entrance.

f) Save the tune file as H2ymmdd.u. Print the Tune page.

10.3.4 EM Adjustment - The Detector Parameters and discriminator voltages must be adjusted at least every three weeks, after the vacuum is restored after having been vented, and after any major maintenance. Using the tuning solution, from the Tune screen, select Tune → Autotune , check the boxes for EM (electron multiplier voltages) and Discriminator (threshold for noise rejection), then click run.

10.3.5 P/A Multiplier Facto Tune r - should be performed prior to each day's use.

a) Aspirate P/A Solution 1+2.

b) From the Tune Screen, select Tune → P/A Factor and click run.

c) The P/A Tuning Factor Report is automatically generated. Assure that a range of elements across the mass range are acceptable. (P/A Tuning does not require all analytes in the set to be calibrated.)

## 11. Sample Analysis

11.1 Sequence

11.1.1 From the ICP_MS Top Screen in the Mass Hunter Software, choose Sequence → Edit Sample Log Table. From the dropdown menu, choose SMPL.

11.1.2 In the METHOD column, choose the method to be run. (Double clicking on any method space will bring up the SELECT METHOD menu.)

11.1.3 In the TYPE column, using the drop down menu, choose a sample type for each sample e. g.. CALBLK, CALSTD, 2-CCV, 2-CCB, Sample.

11.1.4 Enter the calibration standards. Under Dil/Lvl, assign a level for each standard from the dropdown box e. g.: CALBLK is Level 1; the lowest standard is Level 2 and in order until all standard have been assigned a level.

11.1.5 In the VIAL column, assign each standard a position in the autosampler.

11.1.6 List the ICV/CCV, ICB/CCB, PB, LCS's and samples in the sequence table. Include CCSV's, and Low Level Checks when required.

11.1.7 Assign each sample a vial position in the autosampler; enter the dilution factor and any comments.

11.1.8 Import a Data Analysis method by choosing a previous batch that is similar to the batch being run. The DA method may be modified during or after the run if needed.

11.1.9 Click ok, then choose Sequence → Save and enter a file name: mmddyy.s, and click ok.

11.2 Method

11.2.1 From the ICP-MS Top window, check that the method and sequence being used appear at the top, then choose Method → EDIT ENTIRE METHOD.

11.2.2 Check all boxes in the EDIT METHOD dialogue box to review all items.

11.2.2.1 Method Information – enter any comments and click ok.

11.2.2.2 Select QC Items – usually no changes are needed, click ok.

11.2.2.3 Interference Equations – 2008MOD should appear for no gas mode. No equations are used for He or H2 mode.

11.2.2.4 In the Acquisition Mode dialogue box, choose Spectrum Multi-Tune. Select the correct Tune files. The average of three readings is to be used. Make any changes to the acquisition parameters and click ok. It is in this screen that masses are chosen, integration times are set and Modes (no gas, Helium, Hydrogen) are indicated.

11.2.2.5 Peristaltic Pump Program dialogue box – review and make changes if necessary. This program allows for adequate times for sample uptake and rinses between samples. The present settings are:

| | | |
|---|---|---|
| Before Acquisition | | Uptake Speed 0.5 rps |
| | | Uptake time 45 seconds |
| | | Stabilization Time 25 seconds |
| After Acquisition (Rinse Port) | | |
| | | Rinse Speed 0.1 rps |
| | | Rinse time (Sample) 10 seconds |
| | | Rinse time (STD) 10 seconds |
| After Acquisition (Rinse Vial) | | |
| Step 1 | Rinse Vial 1 | Rinse Speed 0.5 rps |
| | Rinse Time | 30 seconds |
| Step 2 | Rinse Vial 2 | Rinse Speed 0.5 rps |
| | Rinse Time | 30 seconds |

Pre-emptive Time 35 seconds

11.2.3 If no major changes are made, save the method under the same name. If major changes are necessary, save the method under a new name.

11.2.4 Go back to the Sequence Tab, click Run, then click Run Sequence on the Start Sequence dialogue box.

11.3 Mass Hunter Data Analysis - From the Batch run window, open the Data Analysis Method Editor.

11.3.1 DA Method Tab - verify that Full Quant analysis and QC check on Full Quant are checked. Spectrum Mode, Count Subtraction except for ISTD and Interference Correction Acq. Defined should be chosen.

11.3.2 Analyte List - check that the correct Elements, Masses and Tune Steps are entered. and that the correct Analyte/ISTD designation has been chosen.

11.3.3 Full Quant – Fill in all values listed for the Calibration Standards and the Internal Standards. The number of Levels and the concentration matching each level must be adjusted on this screen to match those actually being used for the run.

11.3.4 QC Parameters – Verify the QC parameters entered for 2-CCV and 2-CCB, ISTD recovery limits, LDR and RSD.

11.3.5 Return to the Batch run sheet and click on Process Batch to implement any changes. Restart the Sequence, if necessary.

11.3.6 A Results Report page is generated for each determination. QC failures (for ISTD recovery, %RSD, LDR, ICV/CCV Recovery) are flagged.

11.4 The routine sample analysis protocol is as follows:

- Calibration Blank
- Mixed Calibration Standards

- Calibration Blank if required
- Mixed Calibration Standards if required

- ICV
- ICB

- Low Level Check Standards if required

- Prep Blank
- LCS1 & 2

- Environmental Sample
- Matrix Spike
- Environmental Samples

- Calibration Blank if required
- Mixed Calibration Standards if required

OR

- CCV
- CCB
- Low Level Check Standards if required

11.5 During the analytical run the laboratory must comply with the appropriate quality control requirements listed in section 14 of this SOP.

11.6 Determined sample analyte concentrations that are ≥ 110% of the highest standard must be diluted with 2% $HNO_3$ & reanalyzed.

## 12. Data Analysis and Calculations

12.1 Aqueous Samples - undigested

All dilution factors required as a result of dilutions made during analysis are applied at the instrument. All of the aqueous sample results generated from the analysis (in µg/L ) can be reported directly from the instrument.

12.2 Aqueous Samples - digested

All dilution factors required as a result of the digestion procedure or dilutions made during analysis are applied at the instrument. All of the aqueous sample results generated from the analysis (in µg/L) can be reported directly from the instrument.

12.3 All aqueous samples results are manually transcribed to LIMS. Results and are reported to two significant figures and in most cases, are reported down to the standard reporting limits listed in Table 1.

12.4. Non-Aqueous (Sediments and NAPL) Samples

All dilution factors required as a result of dilutions made during analysis are applied at the instrument. Therefore, all of the results, generated from the analysis (in µg/L), can be used directly from the instrument. These "µg/L" results must then be

converted to "mg/Kg" results. The µg/L result is multiplied by the final digestate volume in Liters, usually 0.050 L, and divided by the sample mass in grams, usually 0.50 g (the specific sample volume and mass are recorded in the metals sample preparation log book). For dry weight calculation, the mg/Kg results must be divided by the decimal version of the percent solids, e.g., 90% is 0.90. Refer to SOP G-23 for Percent Dry Solids.

$$Sample\,\mathrm{Re}sult, mg/Kg, (dry\ weight\ basis) = \frac{ug/L \quad x \quad V}{W \quad x \quad (\%\ Solids/100)}$$

where µg/L = Instrument reading (average of three replicates)
V = Final sample volume in liters (e.g. 0.050L)
W = Weight of wet sample in grams (e.g. 0.50g)

All non-aqueous sample results in mg/Kg are manually transcribed to LIMS. All mg/kg results are reported to two significant figures and, in most cases, are reported down to the standard reporting limits listed in Table 1

## 13. Method Performance

An initial demonstration of capability (DOC) must be performed each time there is a significant change in the chemistry of the method, a major modification to an existing instrument, or a new instrument is installed. A DOC is performed by each analyst designated to analyze samples using this method. An annual check must subsequently be performed and documented for each analyst using this method.

### 13.1 Accuracy and Precision

#### 13.1.1 Initial Demonstration of Capability

An initial demonstration of capability study must be conducted for this method for each analyst using this method. The study consists of the analysis of four standards which are from a source independent of the standard curve. The results of the standards must be within the acceptance criteria supplied by the manufacturer or within ±10% if none are specified. The % RSD should be within 20%. The results of the accuracy and precision study (true value, % recovery, standard deviation and % RSD) are maintained by the Quality Assurance Officer for each analyst and are located in the Central Branch File.

#### 13.1.2 Continuing Demonstration of Capability

An annual continuing demonstration of capability study must be performed and

documented. It may consist of either successfully analyzing a PT sample or analyzing 2 sets of AQC standards to within control limits as stated in section 13.1.1. The results of the continuing accuracy and precision study (true value, % recovery, standard deviation and % RSD or final report from the PT provider) are maintained by the Quality Assurance Officer for each analyst and are located in the Central Branch File.

13.2 Method Detection Limit (MDL)

An MDL Study must be conducted for this method. The study is based on the requirements listed in 40 CFR Part 136 Appendix B. Specific procedures for conducting an MDL study can be found in SOP # G-8. The MDL Study is comprised the analysis of seven reagent grade water samples fortified at a level between 2-3x of the detection limit. The results of the MDL determination (true value, average concentration, standard deviation and calculated MDL) are maintained by the Quality Assurance Officer for each method and are located in the Central Branch File.

13.3 Linear Dynamic Range (LDR)

The LDR must be determined by generating a normal linear calibration curve followed by the analysis of successively higher standard solutions. The results of these standard solutions are used to calculate % recovery. This is conducted until the % recovery falls below 90%. The last standard that had a % recovery of at least 90% is identified as the LDR limit. The LDR should be verified when there is a change in analytical performance due to a significant change in instrument hardware or operating conditions. The results of the LDR Study are maintained by the Quality Assurance Officer in the Central Branch File.

13.4 Limit of Quantitation (LOQ)

The Laboratory performs a Limit of Quantitation (LOQ) study on an annual basis for analytes associated with chemistry methods. The validity of LOQ is confirmed by successful analysis of a Laboratory Fortified Blank (LFB) at approximately 2X the reporting limit. The recovery of each analyte is within the ±30% acceptance criteria. After this study is completed, it is reviewed and approved by the Laboratory Management. A summary of all LOQ study performance is maintained in the Laboratory Central File.

## 14. Quality Control

14.1 Calibration Curve

Acceptance Criteria - The calibration curve, generated from a minimum of 2 standard

concentration levels and a blank must have a correlation coefficient ≥0.995.

Corrective Action - If the correlation coefficient is < 0.995, the calibration is disallowed. The analysis must be terminated, and repeated after correcting the problem.

14.2 Initial Calibration Verification (ICV)

Acceptance Criteria

For wastewater compliance monitoring (NPDES) samples only, analyze the same number of Calibration Standards including the calibration blank used in generating the calibration curve immediately following the calibration, after every ten samples and at the end of the sample run. Analysis of the calibration standards immediately following calibration must verify that the instrument is within ±10% of calibration.

Analyze the ICV solution, from a separate identifiable source (different vendor or different lot number) than the calibration standards, immediately following each calibration (ICSV for NPDES samples). Analysis of the ICV must verify that the instrument is within ±10% of calibration.

Corrective Action - If the calibration cannot be verified within the specified limits, analysis must be discontinued; the cause determined and/or in the case of drift the instrument recalibrated.

14.3 Continuing Calibration Verification (CCSV/CCV)

Acceptance Criteria

For wastewater compliance monitoring (NPDES) samples only, analyze the same number of Calibration Standards including the calibration blank used in generating the calibration curve after, after a maximum of every ten samples and at the end of the sample run. Analyses of the Continuing Calibration Standard (CCSV) solutions must be within ±15% of calibration.

For all other programs, analyze the CCV solution, from the same source as that used for the ICV, after a maximum of ten samples and at the end of the sample run. The results of the CCV solution must be within ±20% of the true value.

Corrective Action - If the calibration cannot be verified within the specified limits, re-analyze the CCSV/CCV solution(s). If the results of the second analysis of the CCSV/CCV solution(s) are not within the acceptance limits, the analysis must be discontinued, the cause determined and the instrument re-calibrated. All samples following the last acceptable CCSV/CCV solution(s) must be reanalyzed.

14.4 Initial Calibration Blank/Continuing Calibration Blank (ICB/CCB)

Acceptance Criteria - Analyze a calibration blank immediately following the ICV and after every CCV. All ICB/CCB results must be < |Reporting Limit|. For the NPDES program, Calibration Blank results must be < |Reporting Limit|.

Corrective Action - If the result of the ICB/CCB (Calibration Blank for the NPDES program) is > |Reporting Limit|, the analysis should be stopped, the problem identified, and the ICB/CCB/Calibration Blank reanalyzed. If the ICB/CCB/Calibration Blank results remain >|Reporting Limit| the instrument must be recalibrated.

14.5 Preparation Blank (PB) / Method Blank

Acceptance Criteria - Analyze a PB for each batch of 20 or fewer samples. The PB results must be < |Reporting Limit|.

Corrective Action - If the result of the PB is > |Reporting Limit|, then all associated samples with a concentration of <10X the amount found in the PB should be re-prepared and reanalyzed. If the samples cannot be re-prepared, then all affected sample results must be either 1) qualified accordingly, or 2) the reporting limit is raised to the amount found in the sample. Check with the team leader/section chief to determine which option should be used.

Sample results ≥10X the amount found in the PB are not considered to be affected by the blank contamination or drift, so no corrective action is needed.

14.6 Laboratory Control Sample (LCS) / Laboratory Fortified Blank (LFB)

14.6.1 Aqueous LCS

Acceptance Criteria - Analyze two aqueous LCS/LFB samples with each batch of aqueous samples of 20 or less.

The % recovery of the aqueous LCS must be within ±15% of the true value for NPDES wastewater compliance monitoring samples and within ±20% of the true value for all other environmental samples. The relative percent difference (RPD) of the duplicates should not exceed 20%. Calculate accuracy as percent recovery using the following equation

$$\% \text{ Recovery} = \frac{\text{Average of 2 LCS's} \times 100}{S}$$

Where; LCS = Laboratory Control Sample

s = concentration of analytes added to fortify the LCS solution

14.6.2 Solid LCS

Acceptance Criteria - Analyze two solid LCS samples with each batch of solid samples of 20 or less. Calculate accuracy as percent recovery using the following equation:

$$\% \text{ Recovery} = \frac{\text{Average of 2LCS's, (mg/Kg) X 100}}{\text{True Value, mg/Kg}}$$

The % recovery of the solid LCS must be within ±25% of the true value or within the limits established by the vendor. The relative percent difference (RPD) of the duplicates should not exceed 25%.

Corrective Action for 14.6.1 and 14.6.2 - If the % recovery or %RPD results are outside the required control limits, the affected samples should be re-prepared and reanalyzed. If the samples cannot be re-prepared, then all affected sample results must be qualified accordingly.

14.7 Laboratory Fortified Matrix (LFM)/Matrix Spike(MS) Recovery

Acceptance Criteria - Fortify a known amount of analytes to one sample per matrix per project or a minimum of one MS per batch of 10 or fewer NPDES samples or one MS per batch of 20 or fewer samples for other programs. The LFM/MS aliquot must be a duplicate of the aliquot used for sample analysis. When possible, the concentration should be the same as that added to the aqueous LFB/LCS, but should not exceed the midpoint concentration of the calibration curve. Calculate the percent recovery, corrected for background concentration measured in the unfortified sample, and compare the recoveries to the control limits to the designated matrices:- ±20% for aqueous samples; ±25% for solid samples (soils, sediment, and NAPL); and ±50% for sludge and biological tissue samples. Percent recovery may be calculated using the following equation:

w
h
e
r
e

$$R = \frac{\{Cs - C\} \text{ X } 100}{s}$$

where:

R = percent recovery
Cs = fortified sample concentration
C = sample background concentration

s = conc. equivalent of metal added to sample

Corrective Action - If % recovery of the LFM/MS is outside the required control limits, and the laboratory performance is shown to be in control, the recovery problem encountered is judged to be matrix related, not system related. The native sample result of the sample used to produce the MS must be qualified accordingly.

The % recovery of the LFM/MS is not evaluated when the result of the unfortified sample concentration is ≥1.0X the level used to fortify the sample.

14.8 Internal Standard Responses

Acceptance Criteria - The response of any one internal standard must not deviate from within the range of 60% to 125% of the original response in the calibration blank.

Corrective Action - If deviations greater than these are observed, flush the instrument with the rinse blank and monitor the responses in the calibration blank. If the responses of the internal standards are now within the limit, take a fresh aliquot of the sample, dilute by a further factor of two, add the internal standard and re-analyze. When after flushing the response of the internal standards in the calibration blank are out of limits, terminate the analysis and determine the cause of the drift. Consult the supervisor for further corrective action.

14.9 Low-Level Checks

Acceptance Criteria - Analyze appropriate Low Level Check Standards, (lowest standard for each element of interest at it's reporting limit) immediately following the ICV/ICB and at the end of the run. The %Recovery of the Low Level checks must be within ±30% of the true value for all analytes of interest. (For NPDES Samples the CCSV's serve also as a Low Level check.)

Corrective Action –

If a Low Level check cannot be verified within the specified limits of ± 30% for any analytes of interest, re-analyze the Low Level check solution immediately for those analytes of concern only. If the results of the re-analysis for those analytes fall within the control limits, no further corrective action is required.

If the results of the Low Level re-analysis for those analytes do not fall within the control limits or if the re-analysis would be impossible due to unattended/overnight runs, the reporting limit must be raised the level of the next check standard (ICV/CCV or LCS), as long as the sample project requirements allow. For samples with results > the next check standard (ICV/CCV or LCS), no further actions is

necessary.

For projects where the level of next check standard is not acceptable, the analysis must be discontinued; the cause determined, the instrument re-calibrated and samples re-analyzed. If the samples cannot be reanalyzed, then all affected sample results must be qualified accordingly.

14.10 Triplicate Integrations

Acceptance Criteria - Each analysis consists of three separate integrations or readings. This includes the calibration standards, quality control samples and all associated environmental samples. The average of the three measurements is used for reporting results. The RSD must be $<20\%$ for all results that are $\geq$ the reporting limit.

Corrective Action - If the RSD for a calibration standard or quality control sample is outside the control limits, the analysis must be repeated. If the RSD is still outside the control limits, the analysis must be terminated, and repeated after correcting the problem.

If the RSD for an environmental sample is outside the control limits, and the laboratory performance, i. e. CCV, is shown to be in control, the RSD problem encountered is judged to be matrix related, not system related, and the sample should be qualified accordingly.

## 15. Reporting and Validation

15.1 Reporting Limits - The reporting limits are calculated based on the concentration of the lowest calibration standard analyzed. The reporting limits are matrix and dilution dependent. All results are reported to 2 significant figures. Solid matrices are normally reported in dry weight basis.

15.2 Sample Data Package - The sample data package should include but not be limited to the following:

1. ICP-MS QA/QC Checklist with all relevant information included
2. Copies of Log Book entries of Analysis Run Log; Sample Digestion Log, and if required, Sample Percent Solids Log
3. Calibration Report
4. Summary Analysis Form
5. QC Summary Forms; and
6. Instrument generated sample data

15.3 Laboratory Information Management System (LIMS) - The analyst enters the

data in the LIMS under the appropriate analytical codes.

15.4 Data Validation

The data package is given to the reviewer. The review is done by a peer who was not involved in the analysis. Upon completion of the review, including validation of all the appropriate codes in the LIMS for the particular project(s), the data reviewer will sign and date the ICP-MS QA/QC Checklist.

15.5 Data Records

All project records associated with the data package are filed under one designated project file. All other projects associated with the data package are referenced to this designated project file via a "cross reference form". The "cross reference form" is placed in each of the project files that were associated with the data package.

The data package is placed in the bin identified for the designated project file. The records for this designated project file are filed in our locked record cabinets once all data from the project, e.g., non-metal inorganic data, organic data, microbiology data, etc. has been reviewed by the appropriate staff.

## 16. Pollution Prevention

16.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

16.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

16.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult Less is Better: Laboratory Chemical and Management for Waste Reduction, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N. W., Washington D. C. 20036, (202) 872-4477.

## 17. Waste Management

The USEPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process waste should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the Region 2, SOP G-6, "Disposal of Samples and Hazardous Wastes".

## 18. REFERENCES


- U.S. Environmental Protection Agency. "*Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectroscopy*" Method 200.8, Rev. 5.4(EMMC Version), May 1994.
- U.S. Environmental Protection Agency, Region 2, SOP G-6 "*Disposal of Samples and Hazardous Wastes*"
- U. S. Environmental Protection Agency, Region 2, SOP G-8 "*Laboratory Policy for the Determination of Method Detection Limits (MDLs).*"
- U.S. Environmental Protection Agency, Region 2, SOP G-15 "*Laboratory Definitions and Data Qualifiers*"
- U.S. Environmental Protection Agency, Region 2, SOP C-116 "*Preparation of Aqueous, TCLP Extracts, Soil/Sediment/Sludge, Waste Oil/Organic Solvents, and Biological Tissue Matrices by Block Digestion*"
- U. S. Environmental Protection Agency, Solid Waste 846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 6020A "*Induced Coupled Plasma - Mass Spectrometry*" Laboratory Manual, Revision 1, January 1998.

**Table 1 . Recommended Analytical Isotopes, Additional Masses and Reporting Limits**

| Element of Interest | Isotope | Aqueous Reporting Limit, ug/L | Soil Reporting Limit, mg/Kg |
|---|---|---|---|
| Aluminum | 27 | 20 | 20 |
| Antimony | 121, 123 | 2.0 | 0.2 |
| Arsenic | 75 | 1.0 | 0.1 |
| Barium | 135, 137 | 1.0 | 0.1 |
| Beryllium | 9 | 1.0 | 0.1 |
| Cadmium | 106, 108, 111, 114 | 1.0 | 0.1 |
| Chromium | 52, 53 | 1.0 | 0.1 |
| Cobalt | 59 | 1.0 | 0.1 |
| Copper | 63, 65 | 1.0 | 0.1 |
| Lead | 206, 207, 208 | 1.0 | 0.1 |
| Manganese | 55 | 1.0 | 0.1 |
| Molybdenum | 95, 97, 98 | 1.0 | 0.1 |
| Nickel | 60, 62 | 1.0 | 0.1 |
| Selenium | 77, 82 | 5.0 | 0.5 |
| Silver | 107, 109 | 1.0 | 0.1 |
| Thallium | 203, 205 | 1.0 | 0.1 |
| Thorium | 232 | Not analyzed | Not analyzed |
| Uranium | 238 | Not analyzed | Not analyzed |
| Vanadium | 51 | 1.0 | 0.1 |
| Zinc | 66, 67, 68 | 1.0 | 0.1 |
| | | | |
| Krypton | 83 | | |
| Ruthenium | 99 | | |
| Palladium | 105 | | |
| Tin | 118 | | |

Notes: Isotopes recommended for analytical determination are underlined.
The soil reportimg limit is calculated with the assumption of using 0.5g sample with 100% solids and a 50mL digestion volume.

## Table 2. Recommended Elemental Equations for Data Calculations

| Element | Elemental Equation | Note |
|---|---|---|
| Al | (1.000) ( $^{27}C$) | |
| Sb | (1.000) ( $^{123}C$) | |
| As | (1.000) ( $^{75}C$) - (3.127) [ ( $^{77}C$) - (0.815) ( $^{82}C$)] | (1) |
| Ba | (1.000) ( $^{137}C$) | |
| Be | (1.000) ( $^{9}C$) | |
| Cd | (1.000) ( $^{111}C$) - (1.073) [ ( $^{108}C$) - (0.712) ( $^{106}C$)] | (2) |
| Cr | (1.000) ( $^{52}C$) | (3) |
| Co | (1.000) ( $^{59}C$) | |
| Cu | (1.000) ( $^{63}C$) | |
| Pb | (1.000) ( $^{206}C$) + (1.000) ( $^{207}C$) + (1.000) ( $^{208}C$) | (4) |
| Mn | (1.000) ( $^{55}C$) | |
| Mo | (1.000) ( $^{98}C$) - (0.146) ( $^{99}C$) | (5) |
| Ni | (1.000) ( $^{60}C$) | |
| Se | (1.000) ( $^{82}C$) | (6) |
| Ag | (1.000) ( $^{107}C$) | |
| Tl | (1.000) ( $^{205}C$) | |
| Th | (1.000) ( $^{232}C$) | |
| U | (1.000) ( $^{238}C$) | |
| V | (1.000) ( $^{51}C$) - (3.127) [ ( $^{53}C$) - (0.113) ( $^{52}C$)] | (7) |
| Zn | (1.000) ( $^{66}C$) | |

| | Internal Standards | Note |
|---|---|---|
| Bi | (1.000) ( $^{209}C$) | |
| In | (1.000) ( $^{115}C$) - (1.000) ( $^{118}C$) | (8) |
| Sc | (1.000) ( $^{45}C$) | |
| Tb | (1.000) ( $^{159}C$) | |
| Y | (1.000) ( $^{89}C$) | |

Legend:

C - calibration blank subtracted counts at specified mass.

(1) - correction for chloride interference with adjustment for $^{77}Se$. ArCl 75/77 ratio may be determined from the reagent blank. Isobaric mass 82 must be from Se only and not $BrH^+$.

(2) - correction for MoO interference. Isobaric mass 106 must be from Cd only not ZrO+. An additional isobaric elemental correction should be made if palladium is present.

(3) - In 0.4% v/v HCl, the background from ClOH will normally be small. However, the contribution may be estimated from the reagent blank. Isobaric mass from Cr only not ArC+.

(4) - -allowance for isotopic variability of lead isotopes.
(5) - -isobaric elemental correction for ruthenium.

(6) - some argon supplies contain krypton as an impurity. Selenium is corrected for 82Kr by background subtraction.

(7) – correction for chloride interference with adjustment for 53Cr. ClO 51/53 ratio may be determined from the reagent blank. Isobaric mass 52 must be from Cr only not ArC+.

(8) - isobaric elemental correction for tin.

## Table 3. Common Molecular Ion Interferences in ICP-MS

**Background Molecular Ions**

| Molecular Ion | Mass | Element Interference | Molecular Ion | Mass | Element Interference [a] |
|---|---|---|---|---|---|
| $NH^+$ | 15 | | $^{36}ArH^+$ | 37 | |
| $OH^+$ | 17 | | $^{38}ArH^+$ | 39 | |
| $OH_2^+$ | 18 | | $^{40}ArH^+$ | 41 | |
| $C_2^+$ | 24 | | $CO_2^+$ | 44 | |
| $CN^+$ | 26 | | $CO_2H^+$ | 45 | Sc |
| $CO^+$ | 28 | | $ArC^+$, $ArO^+$ | 52 | Cr |
| $N_2^+$ | 28 | | $ArN^+$ | 54 | Cr |
| $N_2H^+$ | 29 | | $ArNH^+$ | 55 | Mn |
| $NO^+$ | 30 | | $ArO^+$ | 56 | |
| $NOH^+$ | 31 | | $ArOH^+$ | 57 | |
| $O_2^+$ | 32 | | $^{40}Ar^{36}Ar^+$ | 76 | Se |
| $O_2H^+$ | 33 | | $^{40}Ar^{+38}Ar^+$ | 78 | Se |
| | | | $^{40}Ar_2$ | 80 | Se |

[a] method elements or internal standards affected by the molecular ions.

**Matrix Molecular Ions**

| BROMIDE Molecular Ion | Mass | Element Interference | BROMIDE Molecular Ion | Mass | Element Interference |
|---|---|---|---|---|---|
| $^{81}BrH^+$ | 82 | Se | $^{81}BrOH^+$ | 98 | Mo |
| $^{79}BrO^+$ | 95 | Mo | $Ar^{81}Br^+$ | 121 | Sb |
| $^{81}BrO^+$ | 97 | Mo | | | |
| CHLORIDE Molecular Ion | Mass | Element Interference | CHLORIDE Molecular Ion | Mass | Element Interference |
| $^{35}ClO^+$ | 51 | V | $^{37}ClOH^+$ | 54 | Cr |
| $^{35}ClOH^+$ | 52 | Cr | $Ar^{35}Cl^+$ | 75 | As |
| $^{37}ClO^+$ | 53 | Cr | $Ar^{37}Cl^+$ | 77 | Se |
| SULPHATE Molecular Ion | Mass | Element Interference | SULPHATE Molecular Ion | Mass | Element Interference |
| $^{32}SO^+$ | 48 | | $^{34}SOH^+$ | 51 | V |
| $^{32}SOH^+$ | 49 | | $SO_2^+$, $S_2^+$ | 64 | Zn |
| $^{34}SO^+$ | 50 | V, Cr | $Ar^{32}S^+$ | 72 | |
| | | | $Ar^{34}S^+$ | 74 | |
| PHOSPHATE Molecular Ion | Mass | Element Interference | PHOSPHATE Molecular Ion | Mass | Element Interference |
| $PO^+$ | 47 | | $PO_2^+$ | 63 | Cu |
| $POH^+$ | 48 | | $ArP^+$ | 71 | |
| GROUP I, II METALS Molecular Ion | Mass | Element Interference | GROUP I, II METALS Molecular Ion | Mass | Element Interference |
| $ArNa^+$ | 63 | Cu | $ArK^+$ | 79 | |
| | | | $ArCa^+$ | 80 | |

| MATRIX OXIDES* Molecular Ion | Mass | Element Interference | MATRIX OXIDES* Molecular Ion | Mass | Element Interference |
|---|---|---|---|---|---|
| TiO | 62 - 66 | Ni, Cu, Zn | ZrO | 106 - 112 | Ag, Cd |
| | | | MoO | 108 - 116 | Cd |

*Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as method analyte.

EPA

# STANDARD OPERATING PROCEDURE

# PREPARATION OF AQUEOUS, TCLP EXTRACTS, SOIL/SEDIMENT/SLUDGE/SOLID, BIOLOGICAL TISSUE AND OTHER MATRICES BY BLOCK DIGESTION

*Signature and Title*

| | | |
|---|---|---|
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| Peer Reviewed by: | Renee Lettieri<br>Renee Lettieri, Chemist, OICS | 3-12-09<br>Date |
| QA Reviewed by: | Sumy P. Cherukara, Quality Assurance Officer | 3/12/09<br>Date |
| Approved by: | Kim Brandon-Bazile<br>Kim Brandon Bazile, Acting Chief, OICS | 3/13/09<br>Date |
| Approved by: | J. R. Bourbon<br>John R. Bourbon, Acting Chief, Laboratory Branch | 3/13/09<br>Date |

*Annual Review*

| | | |
|---|---|---|
| Reviewed by: | Signature | Date |
| Reviewed by: | Signature | Date |

**U.S. ENVIRONMENTAL PROTECTION AGENCY**
**REGION 2**
**DIVISION OF ENVIRONMENTAL SCIENCE AND ASSESSMENT**
**LABORATORY BRANCH**

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# STANDARD OPERATING PROCEDURE

# PREPARATION OF AQUEOUS, TCLP EXTRACTS, SOIL/SEDIMENT/SLUDGE AND BIOLOGICAL TISSUE MATRICES BY BLOCK DIGESTION

## 1. Scope and Application

1.1 This method is used to digest by DigiBLOC, all environmental samples, with the exception of drinking water. These include aqueous, TCLP extracts, soil/sediment/sludge/solid, and biological tissue. Samples are then analyzed using ICP-AES, SOP #C109 or ICP-MS, SOP # C-112.

1.2 This SOP is based on EPA Methods 200.2, Revision 8.8 and EPA Method 200.8, Revision 5.4.

## 2. Summary of Method

2.1 Aqueous or Aqueous TCLP: A suitable aliquot (usually 50 mL) of a well mixed, aqueous or homogeneous extract sample is accurately measured into a Screw Cap Digestion Vessel and heated on the DigiBLOC at 85° C with $HNO_3$ and HCL until the volume is reduced to 20mL. A watch glass is then placed on the tube and the sample is gently refluxed for an additional 30 minutes. After cooling, the sample is brought up to a known volume, capped and mixed. If needed, the digestates may be filtered.

2.2 Soil/Sediment/Sludge/Solid: A ground mixture of an appropriate amount of a soil/sediment sample, depending on the % Solids, generally a (0.5g to 1.0g sample), is weighed out and placed into the bottom of a 50mL Screw Cap Digestion Vessel where it is digested in 5mL reagent grade water with $HNO_3$ and HCL at 95° C for 30 minutes. After cooling, the sample is brought up to a known volume, filtered, capped and mixed. A correction factor derived from a Percent Solids determination is applied to the final result.

2.3 For biological tissue digestion, the sample is accurately weighed into a Screw Cap Digestion Vessel and digested with $HNO_3$ and 30% $H_2O_2$.

2.4 Samples are then analyzed using ICP-AES or ICP-MS. In all instances, great care must be exercised to avoid contamination.

## 3. Definitions

See SOP#G-15 for definitions.

## 4. Interferences

Samples must be well mixed and as homogenous as possible. Soil/Sediments/ Sludges/Solids must be reduced to as small a particle size as practicable.

## 5. Safety

5.1 The toxicity and carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices, e.g. wear proper protective equipment, safety glasses, gloves, lab coat and working in side hoods whenever possible. Refer to Edison Facility Safety Manual Region II (available on the Region II Intranet), Part 2 – Laboratory Safety and Appendices 13/13A - Chemical Hygiene Plan for specific guidelines.

5.2 Safety guidelines for the DigiBLOC

5.2.1 The DigiBLOC must be grounded and have a clearance of 3 inches on all sides. It must be located in an operable fume hood if the DigiVAC is not available. Do not mount DigiBLOC on a surface of flammable material.

5.2.2 The DigiBLOC must be lifted only from the bottom, not by the top white trim. Acquire assistance to move the unit.

5.2.3 Use caution when working around the instrument during operation. The unit has exposed hot surfaces.

## 6. Apparatus and Materials

6.1 DigiBLOC Digestion System consisting of the Hot Block, with two 24 Position Racks with front and back airfoils,

6.2 DigiPROBE Sample Temperature Controller and probe.

6.3 DigiSET Sample Volume Controller and volume probe. (This equipment is available but not used at the present time.)

6.4 DigiVAC Exhaust System (This equipment is available but not used at the present time.)

6.5 Screw Cap Digestion Vessels & Certified 2um Teflon Filters and plungers.

6.6 Disposable ribbed watch glasses.

6.7 Top loading balance capable of measuring 0.01 gram and disposable spatulas for soil/sediment/sludge/solid digestion.

6.8 Porcelain evaporating dishes (195mL), pestles and standard wooden tongue depressors for drying soil/sediment/sludge/solid for digestion.

6.9 Two re-pipettes capable of dispensing 0.25-5.0mL.

6.10 Three automatic pipettes (1-250µL, 1-1000µL and 1-5mL).

## 7. Reagents and Solutions

All reagents must be of high purity and suitable for trace metal analysis.

7.1 Concentrated Nitric Acid (GFS $HNO_3$, Redistilled or equivalent)

7.2 Concentrated Hydrochloric Acid HCl (GFS HCl, 37% Reagent ACS or equivalent)

7.3 Hydrogen Peroxide, 30%

7.4 Reagent Grade Water

7.5 SPEX CertiPrep Custom Claritas Standard High Check containing 250mg/L (250ppm) of each of the following: Ag, Al, As, B, Ba Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Se, Sb, Si, Sr, Ti, Tl, V, Zn, Sn or equivalent.

7.5.1 ICPMS Low Level Spiking Solution (1000µg/L): Add 400µL of Stock Standard (CAL1) to a 100mL volumetric flask containing approximately 50mL Reagent Grade Water, 1.0mL $HN0_3$ and 0.5mL HCl. Dilute to 100mL with Reagent Grade Water.

7.6 SPEX CertiPrep Custom Multi-element Standard ICV II containing 250mg/L (250 ppm) of each of the following: Al, Ca, Fe, Mg, K, Na, Si or equivalent.

7.7 Soil LCS - Environmental Resource Associates: Trace Metals in Soil or equivalent.

7.8 Biological Tissue LCS - DOLT-4. National Research Council, Canada.or equivalent.

## 8. Sample Collection, Preservation, Storage and Holding Time

8.1 Aqueous: Samples may be collected in plastic or glass. Samples must be preserved to a pH<2 using $HN0_3$. Samples are stored at room temperature and should be digested and analyzed within 6 months of collection.

8.2 Soil/Sediment/Sludge/Solid: Samples may be collected in plastic or glass. Samples must be stored in a refrigerator at 4° C and should be digested and analyzed within 6 months of collection unless stored in a freezer at -20° C.

.3 Biological Tissue: Samples may be collected in plastic or glass containers and must be stored in a freezer at -20°C.

8.4 Hazardous Waste, e.g. drum samples usually have no temperature or holding time requirements.

## 9. Sample Preparation

9.1 Aqueous Sample Preparation

9.1.1 Verify that the pH of the sample is <2 using pH test paper. Record in the Metals Sample pH log book. If the pH is >2, add concentrated $HNO_3$ until the pH is <2, then wait at least 16 hours before rechecking the pH and proceeding with the sample prep.

9.1.2 Transfer 50mL (or other suitable aliquot) from a well mixed, acid preserved sample to a screw cap digestion vessel. Prepare one matrix spike per project for each batch of 10 or fewer samples for DW/NPDES and one matrix spike for each batch of 20 or fewer samples for all other programs. In addition, prepare one Prep Blank and two LCS's for each batch of 20 or fewer samples. Refer to Section 14.1 of this SOP for QC procedure.

9.1.3 Add 0.5mL concentrated $HNO_3$ and 0.5mL of concentrated HCl to each tube.

9.1.4 Insert the tubes into the DigiBLOC for solution evaporation at a pre-tuned temperature of 85 °C. If space permits, leave the outer rows empty. Position the extra blank samples under the exhaust hole of the DigiVAC (if used). Carefully position the DigiPROBE in one tube and the DigiSET volume control (if used) set to 20mL in the other tube. If the DigiVAC is unavailable, carefully place the instrument in a hood and proceed. If the DigiSET is not used, monitor volume

visually.

9.1.5 Close the DigiVAC (if used) lid and turn power on to the DigiVAC, DigiSET (if used) and DigiBLOC.

9.1.6 Reduce volume to approximately 20 mL by gently heating at 85°C. Cap each tube with a disposable ribbed watch glass and reflux for 30 minutes.

9.1.7 Remove from DigiBLOC. Allow to cool. Dilute to 50 mL with Reagent Grade water, cap and mix well. Filter, if necessary. (See Section 9.2.7)

NOTE: - For ICPMS analyses requiring low detection limits, dilute to 25mL after digestion with Reagent Grade Water instead of 50mL

9.2 Soil/ Sediment/Sludge/Solid Preparation

9.2.1 Sample Drying

In general, samples will be prepared according to the following protocol:
Greater than 75% solids: - use 0.50g
Between 40% and 75% solids: - use 1.00g
Below 40% solids - dry according to the method below.

Analysts may use discretion in sample size and drying at 60° C in order to achieve a representative sample for analysis.

9.2.1.1 Pre-Drying Method

This method works best for samples that have a high water content. Evaporating dishes and pestles must be rinsed with 10% $HNO_3$.

Transfer the sample to a 195mL porcelain evaporating dish using a standard tongue depressor and dry at 60°C for a minimum of 12 hours. Cool, then grind with a pestle in the porcelain evaporating dish. Mix well, transfer to a plastic or glass container and store in a refrigerator at 4° C until ready to digest.

These sediment samples require a % Solids determination. See the METALS % SOLIDS LOGBOOK for procedure. % Solids results are reported under SOLA in LIMS. This does not preclude an analysis request for % Solids.

Please note that an alternate approach, due to analyst judgment of the sample(s)/project(s) involved, may be used to determine the amount of sample to be processed. See 9.2.1.2 below.

9.2.1.2 "As Received" Method

This method works best for samples that have a low water content. Samples are digested as received. A % Solids determination is performed using the procedure described in the METALS % SOLIDS LOGBOOK. % Solids results are reported under SOLA in LIMS.

9.2.2 Weigh 0.50g or other suitable aliquot of well mixed sample into a 50 mL Screw Cap Digestion Vessel. In addition, prepare a Prep Blank and two LCS's for each batch of 20 or fewer samples. One Laboratory Fortified Matrix (LFM)/Matrix Spike (MS) is prepared for each matrix per project with at least one MS per batch of 10 or fewer NPDES samples or one MS per batch of 20 or fewer samples for other programs. Refer to Section 14.2 for QC prep.

9.2.3 Under a fume hood, add 5 mL Reagent Grade water, 1.0mL conc. $HNO_3$ and 2.5mL of conc. HCL to the tubes. Keep samples under the hood until any reaction subsides.

9.2.4 Insert the tubes into the DigiBLOC for digestion at a pre-tuned and pre-heated temperature of 95°C. This digestion may also be carried out using the DigiVAC.

9.2.5 Place a disposable watch glass on each tube. If being used, close the DigiVAC lid and turn the power on to the DigiVAC

9.2.6 Heat samples at 95° C for 30 minutes. The DigiBLOC takes about 30 minutes to heat up to temperature.

9.2.7 Filtration is required for soil/ sediment/sludge/solid samples. Adjust the final volume of the digestate to 50mL with Reagent Grade water and mix well. Allow the majority of the suspended matter to settle (possibly over night).then, place the certified 2 um Teflon filter into the top of the tube to be filtered and insert the plunger. Slowly and carefully press the plunger down until it reaches the bottom of the tube. Remove the plunger from the filter and dispose of it. Cap the tube.

As an alternate method for filtration of soil/sediment/sludge/solid samples, label a duplicate set of Screw Cap Digestion Vessels. Rinse Whatman #41 filter paper in disposable funnels with approximately 10mL Reagent Grade water. Place rinsed funnels into duplicate Screw Cap DigestionVessels and transfer corresponding sample. Rinse original tube several times with Reagent Grade water and transfer to new sample tube. Dilute to 50mL with Reagent Grade water, cap and mix well.

9.3 Biological Tissue Digestion: Homogenize the samples. Store samples in the freezer if digestion is delayed, then defrost prior to preparation for digestion. Refer to Section 14.3 of this SOP for QC prep

9.3.1 High Fat Procedure

9.3.1.1 All determinations, including LCS's and MS's and Prep Blank can be done in triplicate unless unforeseeable conditions prevent this. In some cases, the client may require triplicate analyses.

9.3.1.2 Weigh 2.0g homogenized sample into each digestion tube. Choose one sample per batch of 20 to digest in triplicate along with a matrix spike. Weigh 1.0g each of Dolt-4 for duplicate LCS's. Include an empty tube for the Prep Blank.

9.3.1.3 Add 5mL concentrated HN03 and swirl to mix. Cover the tube with a ribbed watch glass and heat at 95° C until sample dissolves and clears and fumes subside. At this point, heat for an additional 15 minutes. See NOTE below.

9.3.1.4 The LCS will foam. Remove from heat at the first sign and allow foaming to subside. Swirl to expedite reaction. Replace on digiBlock and continue digesting. When reaction is complete, (sample dissolves and clears and fumes subside) allow to digest for an additional 15 minutes.

NOTE: For fish tissue higher in fat (a ring of fat will remain after the above digestion), add an additional 5mL of concentrated HNO3. Without replacing the watch glass, reduce the volume to approximately 5mL, heating at 95° C, swirling and checking every 15 minutes. Treat associated LCS's and Prep Blank similarly. Remove and cool tubes. The sample color will be dark yellow. If it is known in advance that the fish tissue sample is high in fat or a sample must be redigested, add 10mL of concentrated HNO3 initially, reduce the volume to 5 mL, heating at 95°C, swirling and checking every 15 minutes.

9.3.1.5 Add 30% H2O2 in 0.5mL increments, max 3mL, heating and swirling in between additions, allowing the mixture to effervesce and subside until the sample becomes totally clear. Add 1 mL H2O2, replace watchglass and digest for 30 minutes more. Remove from digiBLOC and cool. Filter if a fat ring remains in the tube. If not, dilute to 20mL with reagent grade water. Cap securely and mix well. Sample is ready for analysis.

NOTE: The heat generated by some samples will start to melt the digestion tube. Watch heating process closely and at the first sign of melting, which will be residue on the lower inner tube that will not go into solution, remove the tube from the digiBLOC, transfer to a new tube and continue the digestion procedure.

9.3.2 Low Fat Procedure

9.3.2.1 All determinations, including LCS's and MS's and Prep Blank can be done in

triplicate unless unforeseeable conditions prevent this. In some cases, the client may require triplicate analyses.

9.3.2 .2 Weigh 2.0 g finely ground and well mixed sample and 1.0 g LCS (Dolt-4) into a 50 mL DigiBLOC tube. Record actual weights in Prep Book. Be careful not to let tissue stick to sides of tube - aim for the bottom of tube. Also prepare a Prep Blank and weigh an additional tissue sample for the DigiPROBE.

9.3.2.3 Add 5 mL concentrated HNO3 and swirl to mix. Heat gently in the DigiBLOC (tuned to 95° C) with continued swirling. If samples begin to foam, remove from heat until foam subsides. Continue to heat/cool and swirl until samples no longer foam, then digest at 95° C until sample appears clear. A 2 g sample should be clear after about 15 minutes of digestion.

9.3.2.4 Foaming is a more serious problem with the LCS. Dolt -4 will foam copiously when warmed with HNO3. Extreme care is needed in swirling and gently heating until the LCS/$HNO_3$ mixture appears clear. Then allow to digest at 95° C in the DigiBLOC for an additional 15 minutes.

9.3.4.5 After digestion with HNO3 is completed, add 0.5 mL 30% $H_2O_2$ in 0.1 mL portions to each of the tubes, swirling and heating with each addition until any effervescence subsides. It then should be safe to add 0.5 mL portions of the H2O, heating in between additions until the samples become totally clear. After samples are totally clear, add 1 mL additional $H_2O_2$, cover the tubes with a ribbed watch glass and digest for 30 minutes more. Remove from the DigiBLOC, cool and dilute to 20 mL with Reagent Grade water. Cap securely and mix well.

## 10. Instrument Operating Conditions

10.1 DigiBLOC set-up

10.1.1 Power ON - Power switch.

10.1.2 Check Temperature Set-Point by pressing the star button (*). Temperature should be set at 85° C for Aqueous and TCLP extracts, 95° C for soil/sediment/sludge/solid and biological tissue. If the temperature must be changed or the DigiPROBE is either connected or disconnected, the instrument must be tuned.

10.1.2.1 Set temperature by pressing and holding the star button (*) while simultaneously pressing the ↑ (arrow up) or the ↓ (arrow down) button to obtain the desired temperature.

10.1.2.2 Tune DigiBLOC as follows:

– Set desired temperature.

– Hold the ↑ (arrow up) and ↓ (arrow down) keys simultaneously for ≈ 3 seconds to enter program mode. The display will show *tunE*.

–While holding the star button (*), hit the ↑ (arrow up) to reach AESP (the E is actually an upside down F) and then release the star button (*).

– Press and hold buttons simultaneously for 3 seconds until the temperature appears. The system will flash between tunE, AESP and the current temperature.

–When tuning is complete, the system will automatically turn tunE off and display the current temperature only.

10.2 DigiBLOC Shut-down

10.2.1 Power OFF - DigiBLOC and DigiVAC if used

10.2.2 Rinse DigiPROBE with Reagent Grade water and place in a clean empty tube.

## 11. Sample Analysis

Actual sample analysis is carried out using methods SOP #C-109 Trace Metals in Aqueous, Soil/Sediment/Sludge/Solid, Biological Tissue- ICP-AES or SOP #C-112 Trace Elements in Aqueous, Soil/Sediment/Sludge/Solid and Biological Tissue by ICP-MS.

## 12. Data Analysis and Calculations

Calculations are not done as part of this method. All weights and dilutions are recorded in the Metals Sample Prep Log Book

## 13. Method Performance

Method performance is evaluated as part of methods SOP #C-109 Trace Metals in Aqueous, Soil/Sediment/Sludge/Solid, and Biological Tissue by ICP-AES or SOP #C-112 Trace Elements in Aqueous, Soil/Sediment/Sludge/Solid and Biological Tissue by ICP-MS.

## 14. Quality Control

14.1 Aqueous Quality Control

14.1.1 One Prep Blank (PB) is prepared for every batch of 20 or fewer samples. The PB is prepared by transferring 50mL of Reagent Grade water into a 50mL Screw Cap Digestion Vessel and adding 0.5mL concentrated $HNO_3$ and 0.5mL of concentrated HCl to each tube.

14.1.2 Two LCS's (Laboratory Control Samples) are prepared for every batch of 20 or fewer samples for DW/NPDES and all other programs. These LCS's are made by pipetting 40µL of (CAL I) SPEX CertiPrep Custom Claritas Standard (250PPM) and 1000µL of SPEX CertiPrep Custom Multi-element Standard ICV II (250PPM) into 50mL Screw Cap Digestion Vessels containing 50 mL Reagent Grade water, 0.5mL concentrated $HNO_3$ and 0.5mL of concentrated HCl. Dilute to 50 mL after digestion..

NOTE: For ICPMS: Low Level LCS Add 1mL of ICPMS Low Level Spiking Solution (1000µg/L) to 50mL Screw Cap Digestion Vessels containing 50mL Reagent Grade Water, 0.5 mL $HNO_3$ and 0.5mL HCl in duplicate, per batch of 20 or fewer samples for DW/NPDES and two LCS's for each batch of 20 or fewer samples for all other programs). Dilute to 50 mL after digestion.

14.1.3 Fortify a known amount of analytes to one sample per matrix per project at a minimum of 10% for DW/NPDES or 5% for all other programs. The MS is prepared by adding 40µL of (CAL I) SPEX CertiPrep Custom Claritas Standard (250PPM) and 1000µL of SPEX CERTIPREP Custom Multi-element Standard ICV II (250PPM) to a Screw Cap Digestion Vessel containing 50 mL of a duplicate environmental sample, 0.5mL concentrated $HNO_3$ and 0.5mL of concentrated HCl. Dilute to 50 mL after digestion.

NOTE: For ICPMS: Low Level Matrix Spike - Add 0.5mL of ICPMS Low Level Spiking Solution (1000µg/L) to 50mL Screw Cap Digestion Vessel containing 50mL of a duplicate environmental sample, 0.5 mL $HNO_3$ and 0.5mL HCl and dilute to 50 ml after digestion. One Matrix Spike (MS) is prepared for each matrix per project per batch of 10 or fewer samples for DW/NPDES and one for each matrix per project per batch of 20 samples for all other programs

The concentration chosen for the Matrix Spike can be varied, but should not exceed the midpoint concentration of the calibration curve.

14.2 Soil/Sediment/Sludge/Solid Quality Control

14.2.1 One Prep Blank should be prepared for every batch of 20 or fewer samples. The PB is prepared by transferring 5mL of Reagent Grade water into a 50mL Screw Cap Digestion Vessel and adding 1.0mL $HNO_3$ and 2.5mL HCl. Dilute to 50 mL after digestion.

14.2.2 Two LCS's are prepared for every batch of 20 or fewer samples. These LCS's are made by weighing 0.5g of ERA's Trace Metals in Soil into a 50mL Screw Cap Digestion Vessel and adding 5mL of Reagent Grade water to wash down the sides of the tube, 1.0mL $HNO_3$ and 2.5mL HCl. Dilute to 50 mL after digestion.

14.2.3 Fortify a known amount of analytes to one sample per matrix per project or a minimum of 5% whichever is greater. The MS is prepared by adding 40µL of (CAL I) SPEX CertiPrep Custom Claritas Standard (250PPM) and 1000µL of SPEX CERTIPREP Custom Multi-element Standard ICV II (250PPM) to a 50mL Screw Cap Digestion Vessel containing 0.5g of a duplicate environmental sample, 5mL of Reagent Grade water, 1.0 mL $HNO_3$ and 2.5mL HCl.

The concentration chosen for the Matrix Spike can be varied, but should not exceed the midpoint concentration of the calibration curve:

14.3 Biological Tissue Quality Control

14.3.1 One Prep Blank is prepared for every batch of 20 or fewer samples. See section 9.3.1.2 for high fat digestion and 9.3.2.2 for low fat digestion.

14.3.2 Two LCS's are prepared for every batch of 20 or fewer samples. See Section 9.3. 1.2

14.3.3 One sample per batch of 20 is digested in triplicate. See Section 9.3.1.2 for high fat digestion and 9.3.2.2 for low fat digestion.

14.3.4 One matrix spike (MS) of the triplicate sample per batch of 20 is also digested. See Section 9.3.1.2 for high fat digestion and 9.3.2.2 for low fat digestion.

The concentration chosen for the Matrix Spike can be varied, but should not exceed the midpoint concentration of the calibration curve.

## 15. Reporting and Validation

Copies of all Log Book entries (Sample Preparation, Percent Solids) are included in the final

data packages.

## 16. Pollution Prevention

16.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the USEPA recommends recycling as the next best option.

16.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

16.3 For information about pollution prevention that may be applicable to laboratories, consult *"Less is Better: Laboratory Chemical Management for Waste Reduction"*, available from the American Chemical Society's Department of Government Regulations and Science Policy, 115 16$^{th}$ Street N.W., Washington D.C 20036, (202)872-4477.

## 17. Waste Management

The USEPA requires that laboratory waste management practice be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any water discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the Region 2 SOP #G-6, "Disposal of Samples and Hazardous Wastes".

## 18. References

1. EPA Method 200.2, Revision 2.8.
2. SW846 3010A
3. DigiBLOC 3000 Digestion System Operation Manual
4. DigiVAC Operation Manual

**ADDENDUM #1**

## SAMPLE PREPARATION FOR DISSOLVED SILICA AND PARTICULATE SILICA

1. **Scope and Application**

   1.1 For Dissolved Silica, a 50mL aliquot of well mixed sample is accurately measured into a 50mL screw cap vessel and heated on the digiBLOC at 85° C with $HNO_3$ and HCl until the volume is reduced to 20mL. A ribbed watch glass is then placed on the tube and the sample is gently refluxed for an additional 30 minutes. After cooling, the sample is brought up to 50mL, capped and mixed. If needed, the digestates may be filtered.

   1.2 For Particulate Silica (filter containing sample filtrate), the filter is initially digested in 5mL reagent grade water and 1mL each of $HNO_3$ and HCl, heated on the digiBLOC at 95° C for 45 minutes, cooled and diluted to 50mL. It is then heated on the digiBLOC at 85° C with additional $HNO_3$ and HCl until the volume is reduced to 20mL. A ribbed watch glass is then placed on the tube and the sample is gently refluxed for an additional 30 minutes. After cooling, the sample is brought up to 50mL, capped and mixed. These digestates will need filtering.

2. **Apparatus And Materials**

   2.1 DIGIbloc 3000
   2.2 Millepore Filters, TYPE HNWP, 0.45µm
   2.3 Screw Type Digestion Vessels
   2.4 Ribbed Watch Glasses
   2.5 Filtermate
   2.6 Disposable Forceps

3. **Reagents And Solutions**

   3.1 Concentrated $HNO_3$
   3.2 Concentrated HCl
   3.3 25 PPM Silica - 1mL of 250 PPM Si to 10mL.

4. Procedure

NOTE: For contamination purposes, no glass may come in contact with the samples. This also applies to the addition of the acids – no dispensers.

4.1 Dissolved Silica – See Section 9.1 of this SOP.

4.2 Particulate Silica

4.2.1 Allow filters to defrost. (They will be placed in a digestion tube after filtration so they will be ready for digestion. No other transfer is necessary.)

4.2.2 Prepare 1 tube for Prep Blank and 2 tubes for LCS's by placing a blank filter via disposable forceps in each one.

4.2.3 Add 5mL of reagent grade water to each tube

4.2.4 Using a disposable forceps, gently fold and ease the filter to the bottom of the tube, submerging it as much as possible, being very careful not to disturb the particulates on the sample filter paper.

4.2.5 Add 1mL each of concentrated $HNO_3$ and concentrated HCl to the tubes and 200uL of 25PPM Silica to both LCS's.

4.2.6 Add disposable watch glasses to all tubes and heat @ 95° C on the digiBLOC for 45 minutes.

4.2.7 Cool to touch and dilute to 50 mL

4.2.8 Proceed with section 9.1 of this SOP.

**ADDENDUM #2**

## SAMPLE PREPARATION FOR LEAD ANALYSIS FOR GHOST WIPES

### 1. SCOPE AND APPLICATION

1.1 This method covers the preparation of lead wipes specifically the Ghost Wipe from Environmental Express. The sample is heated in the presence of Nitric and Hydrochloric acids to dissolve the wipe and all lead compounds into solution. The analysis is to be performed by Inductively Coupled Plasma technique (ICP).

### 2. APPARATUS AND MATERIALS

2.1 digiBLOC 3000
2.2 Ghost Wipes
2.3 Screw Cap Digestion Vessels
2.4 Ribbed Watchglasses
2.5 Filtermate (optional- if sample does not completely dissolve)
2.6 Disposable Forceps

### 3. REAGENTS

3.1 Concentrated Nitric Acid ($HN0_3$)
3.2 Concentrated Hydrochloric Acid (HCL)
3.3 Solution containing 400mL reagent grade water, 80mL concentrated $HN0_3$ and 80mL concentrated HCl.

### 4. PROCEDURE

4.1 Use disposable forceps to transfer the wipes from the sample container to the digestion vessel. It is recommended that the wipe is sent to the sampling area with a cup and wipe therefore no transfer steps are encountered. The wipe must be transported in a rigid walled container according to the sampling procedure ASTM E1728.

4.1 Rinse sample container with 2 separate aliquots, 7mL each, of the above acid solution and transfer to digestion vessel. Cap with ribbed watchglass.

4.2 Place digestion tubes in digiBLOC, tuned to 95°C, and begin heating. This slows down any possible foaming. If the tube is placed in a pre-heated digiBLOC, the mixture may turn black, then brown and quickly foam out of the tube. The samples must be watched closely and should signs of foaming begin, be removed immediately and allowed to settle down before proceeding with the digestion.

4.3 After effervescence subsides, heat the sample for 45 minutes at 95° C.

4.4 Remove from the digiBLOC and allow the samples to cool. Dilute each to 50mL and proceed with the 2nd part of this digestion. Follow section 9.1 of this SOP (C-116) Aqueous Sample Preparation. Add 0.5mL of concentrated $HN0_3$ and 0.5mL of concentrated HCl. Place samples in a dgiBLOC, tuned to 85° C. Heat until volume reduces to approximately 20mL. Cap with ribbed watch glass and reflux for 30 minutes. Remove samples from digiBLOC and cool. Dilute each to 50mL, cap and shake. If excess amounts of undigested material remains, filter the sample using a 2.0u Teflon Filtermate.

4.5 Actual sample analysis is carried out using methods SOP #C-109 Trace Metals in Aqueous, Soil/Sediment/Sludge/Solid, Biological Tissue- ICP-AES or SOP #C-112 Trace Elements in Aqueous, Soil/Sediment/Sludge/Solid and Biological Tissue by ICP-MS.

NOTE: This method was adapted for use with the Environmental Express HotBlock and strictly following the HUD (Housing & Urban Development) guidelines for Lead in Dust Wipes Appendix A-5.0 which references NIOSH 7082.

EPA

# STANDARD OPERATING PROCEDURE

# DETERMINATION OF MERCURY IN AQUEOUS, SOIL/SEDIMENT AND BIOLOGICAL TISSUE MATRICES BY THERMAL DECOMPOSITION, AMALGAMATION, AND ATOMIC ABSORPTION SPECTROPHOTOMETRY

*Signature and Title*

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*Annual Review*

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| Reviewed by: | _____<br>Signature | _____<br>Date |

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION 2
DIVISION OF ENVIRONMENTAL SCIENCE AND ASSESSMENT
LABORATORY BRANCH

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# DETERMINATION OF MERCURY IN AQUEOUS, SOIL/SEDIMENT AND BIOLOGICAL TISSUE MATRICES BY THERMAL DECOMPOSITION, AMALGAMATION, AND ATOMIC ABSORPTION SPECTROPHOTOMETRY

## 1. Scope and Application

1.1 This SOP is applicable for the determination of Total Mercury in aqueous, soil/sediment, and biological tissue matrices environmental samples without chemical pretreatment.

1.2 This SOP is not applicable to the analysis of NPDES or Drinking Water regulatory compliance monitoring samples.

NPDES regulatory compliance monitoring samples must be determined by SOP #C-110: Determination of Mercury in Aqueous, Soil/Sediment, Waste Oil/Organic Solvents, TCLP Extracts and Biological Tissue Matrices by Cold Vapor Atomic Absorption Spectrometry.

Drinking Water compliance monitoring samples must be determined by SOP # DW-7: Determination of Mercury in Drinking Water by Cold Vapor Atomic Absorption Spectrometry.

Samples high in organic or volatile material require special handling in order to be analyzed by this method. See Section 12.

1.3 The standard reporting limit for soil/sediment is 0.01mg/kg. This is based on the concentration of the lowest calibration standard analyzed and assumes 100% solids for solid matrices.

1.4 This SOP is based on EPA Method 7473, Revision 0, January 1998.

1.5 All analysts must satisfactorily perform an initial demonstration of capability (DOC) by meeting the method performance criteria in section 13, prior to performing sample analysis using this SOP.

## 2. Summary of SOP

2.1 Controlled heating in an oxygenated decomposition furnace is used to liberate mercury from samples in the instrument. The sample is dried and then thermally and chemically

decomposed within the decomposition furnace. The decomposition products are carried by flowing oxygen to the catalytic section of the furnace. Here oxidation is completed and halogens and nitrogen/sulfur oxides are trapped. The remaining decomposition products are then carried to an amalgamator that selectively traps mercury. After the system is flushed with oxygen to remove any remaining gases or decomposition products, the amalgamator is rapidly heated, releasing mercury vapor. Flowing oxygen carries the mercury vapor through absorbance cells positioned in the light path of a single wavelength atomic absorption spectrophotometer. Absorbance (peak height or peak area) is measured at 253.7 nm as a function of mercury concentration.

2.2 The typical working range for this method is 1.0 - 500 ng. The mercury vapor is first carried through a long pathlength absorbance cell and then a short pathlength absorbance cell. (The lengths of the first cell and the second cell are in a ratio of 10:1.). The same quantity of mercury is measured twice, using two different sensitivities, resulting in a dynamic range that spans at least four orders of magnitude.

2.3 The instrument detection limit (IDL) for this method is 0.01 ng total mercury.

## 3. Definitions

3.1 Thermal Decomposition: Partial or complete degradation of sample components using convection and conduction heating mechanisms, resulting in the release of volatile components such as water, carbon dioxide, organic substances, elements in the form of oxides or complex compounds, and elemental gases.

3.2 Amalgamation: The process by which mercury forms a metal alloy with gold.

3.3 Amalgamator: A system composed of gold particles at a high surface area to volume ratio for the purpose of amalgamating mercury vapor.

3.4 Primary Calibration: A complete calibration of the instrument’s working range. This calibration is performed initially and when any significant instrumental parameters are changed. For example, in this method a primary calibration should be performed after the decomposition tube, amalgamator, or oxygen tank is replaced.

3.5 Daily Calibration Verification: A calibration performed with minimal standards to ensure that the primary calibration is valid. For example, when two standards within the range of interest are analyzed and agree within 10% of their true value, the primary calibration is assumed to be valid.

3.6 Memory Effects: Mercury vapor may remain in the decomposition tube, amalgamator, or absorbance cells and be released in a subsequent analysis resulting in a positive bias. For example, this may result when a low concentration sample is analyzed after a sample of high mercury content.

3.7 Sample Boat: The non-amalgamating thermally stable vessel used for containment and transport of the solid or liquid sample for thermal decomposition.

## 4. Interferences

Memory effects between analyses may be encountered when analyzing a sample of high mercury concentration ($\geq$ 400ng) prior to analyzing one of low concentration ($\leq$ 25 ng). To minimize memory effects, analyze the samples in batches of low and high concentrations, always analyzing those of low concentration first. If this batching process cannot be accomplished, several blank analyses may be required following the analysis of a high level sample to limit memory effects.

## 5. Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices, e.g. wear proper protective equipment, safety glasses, lab coat, and working inside hoods whenever possible. Refer to Edison Facility Safety Manual Region II (available on the Region II Intranet), Part 2 - Laboratory Safety and Appendices 13/ 13A – Chemical Hygiene Plan for specific guidelines.

5.2 Many mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Extreme care must be exercised in the handling of concentrated mercury reagents. Concentrated mercury reagents should only be handled by analysts knowledgeable of their risks and of safe handling procedure.

## 6. Apparatus and Equipment

6.1 Milestone Direct Mercury Analyzer 80 - includes decomposition tube, decomposition and catalyst furnaces, amalgamator, cuvettes, heating unit, low pressure mercury vapor lamp, 254 nm installed with 9 nm band width interference filter, silicon UV photodetector, 40-position autosampler, and instrument controller with software, or equivalent.

6.2 High purity oxygen gas - High purity oxygen should be interference and mercury free. If the oxygen is possibly contaminated with mercury vapor, a gold mesh filter should be inserted between the gas cylinder and the mercury analysis instrument to prevent any mercury from entering the instrument.

6.3 Compatible 4 place Analytical Balance with interface cable (9-pinsub-D).

6.4 Nickel boats for soil and fish tissue standards and samples, and quartz boats for aqueous standards and samples.

6.5 Amber Glass Bottles - Amber Glass Bottles of 25mL and 50mL capacity are used for the preparation of both stock calibration and reference standards and blanks.

6.7 Motorized Microliter Pipettes and Tips - For dispensing microliter volumes of calibration and reference standards and samples. Rainin Instrument Co., Inc., or equivalent. volume measurement devices.

## 7. Reagents and Solutions

7.1 Reagents

All reagents must be of high purity and suitable for Mercury analysis.

7.1.1 Reagent grade water - ASTM Type 1 Water

7.1.2 Hydrochloric Acid (HCl), Concentrated: Reagent grade GFS Chemicals, Inc. or equivalent.

7.1.3 Nitric Acid ($HN0_3$), 5%: Redistilled grade GFS Chemicals, Inc. or equivalent.

7.1.4 Common white flour

7.1.5 Soil LCS - Environmental Resource Associates: Trace Metals in Soil or equivalent.

7.1.6 Biological Tissue LCS - DOLT-4. National Research Council, Canada or equivalent.

7.2 Solutions

Only high grade standard solution that conform to the ACS specifications should be used whenever possible.

7.2.1 Mercury Stock Standard (CAL)-AbsoluteGrade NIST Traceable Mercury Analytical Reference Material 2% $HNO_3$, 1000mg/L (CAL) or equivalent. (For other vendors and/or concentrations, dilutions may be adjusted to yield 1.0PPM and 0.1PPM Hg working standards.)

7.2.1.1 Hg DMA Stock Calibration Standard (10PPM)) - pipet 0.5mL of Absolute Grade NIST Traceable Mercury Analytical Reference Material 2% $HNO_3$, 1000mg/L (CAL) into a 50 mL amber glass bottle containing 5mL of concentrated HCl and 44.5mL of reagent grade water and mix. Prepare fresh every 6 months.

7.2.1.2 1.0 PPM Calibration Standard - pipet 2.5mL of Hg DMA Calibration Stock Standard (10 PPM) into a 25 mL amber glass bottle containing 2.5mL of concentrated HCl and 20mL of reagent grade water and mix. Prepare fresh every 28 days.

7.2.1.3 0.1 PPM Calibration Standard - pipet 0.25mL of 10PPM Calibration Standard into a 25 mL amber glass bottle containing 2.5mL of concentrated HCl and about 22.25mL of reagent grade water and mix. Prepare fresh every 28 days.

7.2.2 Mercury Stock Standard (ICV)-AbsoluteGrade NIST Traceable Mercury Analytical Reference Material 2% $HNO_3$, 1000mg/L (ICV) or equivalent. (For other vendors and/or concentrations, dilutions may be adjusted to yield 1.0PPM and 0.1PPM Hg working standards.) This standard must be of a different lot than that used for the Calibration curve.

7.2.2.1 Hg DMA Stock ICV (10PPM) - pipet 0.5mL of Absolute Grade NIST Traceable Mercury Analytical Reference Material 2% HNO3, 1000mg/L (ICV) into a 50mL amber glass bottle containing 5mL of concentrated HCl and about 44.5mL of reagent grade water and mix. Prepare fresh every 6 months.

7.2.2.2 1.0 PPM ICV - pipet 2.5mL of Hg DMA Stock ICV (10PPM) into a 25 mL amber glass bottle containing 2.5mL of concentrated HCl and 20 mL of reagent grade water and mix. Prepare fresh every 28 days.

7.2.2.3 0.1 PPM ICV - pipet 0.25mL of 10 PPM ICV into a 25 mL amber glass bottle containing 2.5mL of concentrated HCl and 22.25mL of reagent grade

water and mix. Prepare fresh every 28 days.

7.2.3 10% HCl - pipet 2.5mL of concentrated HCl into a 25mL amber glass bottle containing 22.5mL reagent grade water. This solution is used for the analysis of Calibration Blanks, ICB's, CCB's and Prep Blanks.

7.2.4 7% HNO3. - pipet 1.75mL of concentrated $HNO_3$ into a 25mL amber glass bottle containing 23.25mL reagent grade water. This solution is used for the catalyst tube conditioning procedure.

## 8. Sample Collection, Preservation, Storage and Holding Time

8.1 Sample Collection - Sample must be collected in plastic or glass containers.

8.2 Preservation

8.2.1 Aqueous - these samples are preserved using concentrated nitric acid. The preservation is performed either a) in the field at the time of collection, or b) in the Laboratory, within five business days. If the samples are preserved in the Laboratory, the samples must be held for sixteen hours after acidification and then verified at a pH <2 prior to sample processing. If the sample pH is verified to be at a pH >2 after the sixteen hours, additional nitric acid must be added and the sample held for an additional sixteen hours until verified to a pH <2.

8.2.2 Soil/Sediment/Sludge samples - These samples are stored in a refrigerator at $\leq 4^{\circ}C$.

8.2.3 Biological Tissue samples - these samples are preserved in ice. The samples are stored at $\leq -20^{\circ}C$.

8.3 Sample Storage

8.3.1 Aqueous samples that have been preserved at a pH of <2 are stored at room temperature. See 8.2.1.

8.3.2 Soil/Sediment/Sludge samples are stored in a refrigerator at $\leq 4^{\circ}C$.

8.3.3 Biological Tissue samples are stored at $\leq -20^{\circ}C$.

8.4 Holding Time

8.4.1 Aqueous samples must be prepared and analyzed within 28 days of collection.

8.4.2 Soil/Sediment/Sludge samples must be prepared and analyzed within 28 days of collection.

Note: If soil/sediment samples are stored at ≤-20°C, the holding time is extended. The samples must be prepared and analyzed within 12 months of collection.

8.4.3 Biological Tissue samples must be analyzed within 12 months of collection.

## 9. Sample Preparation

9.1 Remove samples from refrigerator and allow to reach room temperature prior to analysis.

9.2 Because of the small weight required for analysis, soil/sediment samples must be dried, ground and mixed well to ensure that a homogeneous sample is analyzed.

9.2.1 Transfer the sample to a 195mL porcelain evaporating dish using a standard tongue depressor and dry at 60$^{o}$ C for a minimum of 12 hours. Cool, then grind with a pestle in the porcelain evaporating dish. Mix well, transfer to a plastic container and store in a refrigerator at 4$^{o}$C until ready to analyze. If samples cannot be analyzed at this time, they must be frozen at ≤-20°C until analysis can be performed.

9.2.2 These sediment samples require a % Solids determination. See the METALS % SOLIDS LOGBOOK for procedure. % Solids results are reported under SOLA in LIMS. This does not preclude an analysis request for % Solids.

## 10. Instrument Operating Conditions

10.1 Turn on the High purity oxygen gas tank. Open oxygen toggle switch by the instrument and adjust the gas pressure to 65 pounds per square inch (psi) to start the flow through the instrument.

10.2 Use the On/Off button to start the DMA-80. Allow 30 minutes for warm-up.

10.3 In the login box, using the stylus, select User (analyst's name) and hit the appropriate keys on the touchpad to enter password and then hit O.K. icon. The main screen of the terminal will appear.

10.4 Hit the DMA-80 Measurement Icon. Four Tab directories will appear at the top of the screen. Hit Meth. Here methods can be created, stored, changed, deleted and loaded. Choose method training.m80 from drop-down menu.

10.5 Hit the Calibr. Tab to move to the calibration screen. Select current primary calibration from drop-down menu. (e.g. 070709 CALABRATION)

10.6 Hit MEAS. Tab to move to the measurement screen.

10.6.1 Hit the clear page icon (third from left on bottom of screen) to clear existing data. Hit the save icon (second from right on bottom of screen) to save this new, blank, file under a new file name.

10.6.2. Double hit the empty name space. A keyboard will appear. Using the stylus, "type" the new data file name (e.g. 091409 REMAP #08070008) and hit OK. Then hit the save icon (right tab at the bottom right of the screen). A new, blank, measure file to add data to has now been created.

## 11. Calibration

NOTE: - Prior to setting up the primary calibration, perform the following catalyst tube conditioning step: Analyze 5 replicates of 100mg common white flour followed by 5 replicates of 100uL 7% $HN0_3$, 2 times.

11.1 Primary calibration: A complete calibration of the instrument's working range is performed initially and when any significant instrumental parameters are changed. For example, a primary calibration should be performed after the decomposition tube, amalgamator, or oxygen tank is replaced. This primary calibration may be used repeatedly until either the 10 ng ICV or the 250ng ICV fall above or below their 10% true value.

11.1.1 Using the working standards described in section 7.2, place the quartz boats in the auto sampler and pipet the volumes listed below for each standard, in duplicate:

| | |
|---|---|
| 0 | 100 uL of 10% HCl |
| 1 ng | 10 uL of 0.1 PPB |
| 5ng | 50 uL of 0.1 PPB |
| 10 ng | 100 uL of 0.1 PPB |
| 15 ng | 150 uL of 0.1 PPB |
| 20 ng | 200 uL of 0.1 PPB |
| 40 ng | 40 uL of 1.0 PPM |
| 100 ng | 100 uL of 1.0 PPM |
| 250 ng | 250 uL of 1.0 PPM |
| 500 ng | 500 uL of 1.0 PPM |

11.1.2 Instrument set-up – see 10.1 – 10.4. The instrument will be warmed up and a method chosen.

11.1.3 Open the "Calibration" screen and press the 'empty page' icon to create a new calibration file. Name the calibration file (e.g. 100809calcurve) and exit the screen.

11.1.4 In the "Measurement" screen, select the first sample position.

11.1.5 Enter standard name in Sample Name column.

11.1.6 Enter standard volume (need to specify units) in Weight column.

11.1.7 Click 'Calibration' from the drop down menu under the 'State' column.

11.1.8 Add ng of working standard used in ng column – the actual standard value will then appear in the ng column.

11.1.9 Enter any number for the ng of blank in the ug/kg column. Even though the value should be zero, the software requires a real number.

11.1.10 Repeat above steps for the remaining standards. Name this data file (e.g. 100809calcurve) and save.

11.1.11 After the last standard is analyzed, the screen will exhibit the actual plotted curve. Select the square fit for the low curve and then, the high curve. If each has a corr. coef. of >0.995 then hit the save tab 2x and answer yes to the overwrite question. This completes the primary calibration procedure.

11.2 Daily Calibration Verification: - Three blanks (Blank Check) containing 100uL of 10% HCl are initially analyzed to ensure that there is no residual carryover in the system.

Then a 1 ng low level standard (10uL of 0.1ppm Hg) is analyzed. This is followed by a low (10ng Hg) and a high (250ng) concentration ICV standard. Three more blanks are analyzed, again, to ensure that there is no residual carryover in the system.

## 12. Sample Analysis

NOTE: When working with highly organic samples it is recommended that sample size be kept under 250mg. When working with highly volatile samples (ex. samples with boiling points lower than 100$^{o}$C) Milestone Inc. recommends using a longer drying time (100 – 200 sec) and lower drying temperature (50$^{o}$C). It is also recommended that a bed of sand or alumina powder be placed inside the boat. Adding the sample to this bed will create a larger heating surface which would minimize the occurrence of an uncontrollable exothermic reaction.

12.1 In the **Meas.,** tab hit the **Result** folder. Hit **autosampler automatic mode** tab (fourth tab from lower left on display screen) for continuous analysis (yellow icon). When clicked again, icon turns to gray, indicating single analysis.

12.2 Hit the add data tab (first tab from lower left on display screen) to actually begin the id weight file.

12.3 Double hit the empty sample name column space. A keyboard will appear. Using the stylus, "type" the sample/standard name and hit OK. Double hit the empty Weight column space next to the sample id.

12.3.1 For aqueous, enter the volume added and pipet that volume into a quartz boat.

12.3.2 For soil/fish tissue, weigh the sample in a nickel boat (except for the matrix spike sample – use the quartz because of the aqueous spike addition) on the analytical balance interfaced with the DMA-80, and press the print button on the balance. The weight will be automatically printed in the weight column or may be typed in manually.

12.3.3 Hit the add data tab again to add additional lines for additional sample/standard/blanks identification

12.3.4 All weights must be entered for all sample id's before the instrument can start analyzing. However, additional samples and weights can be added, after analysis has begun.

Follow the sample analysis protocol:

1. Blank Check - pipet 100 uL of 10% HCl into quartz boat
2. Blank Check: - pipet 100 uL of 10% HCl into quartz boat
3. Blank Check: - pipet 100 uL of 10% HCl into quartz boat
4. Low Check, 1 ng: - pipet 10 uL of 0.1 PPM CAL into quartz boat
5. ICV 10 ng -pipet 100 uL of - 0.1 PPM ICV into quartz boat
6. ICV 250 ng - pipet 250 uL of 1.0 PPM ICV into quartz boat
7. Blank Check -pipet 100 uL of 10% HCl into quartz boat
8. Blank Check - pipet 100 uL of 10% HCl into quartz boat
9. Blank Check (ICB): - pipet 100 uL of 10% HCl into quartz boat
10. LCS-1: - Weigh a sample to contain not more than 500 ng Hg into nickel boat (e.g.<64mg ERA SRM #D059-540, actual weight will vary with each lot)
11. LCS-2: - Repeat as in #10
12. Environmental Sample: weigh 250mg-500mg for unknown samples into nickel boat
13. Environmental Sample MS: same as above + 100 uL of 0.1 PPM CAL into quartz boat
14. Environmental Samples - see #16
15. CCV 10 ng:- pipet 100 uL of 0.1 PPM CAL into quartz boat - repeated every 10 samples and at end of run
16. CCB: -pipet 100 uL of 10%HCl into quartz boat – repeated every samples and at end of run
17. LOW CHECK 1 ng:- pipet 10 uL of 0.1 PPM CAL into quartz boat
18. ICV 250 ng - pipet 250 uL of 1.0 PPM ICV into quartz boat
19. Blank Check: - pipet 100 uL of 10%HCl into quartz boat
20. Blank Check: - pipet 100 uL of 10%HCl into quartz boat
21. Blank Check: - pipet 100 uL of 10%HCl into quartz boat

12.4 Ensure that the concentration column is in the correct units. ug/kg = ppb and mg/kg = ppm.

12.5 Hit LINKS tab to ensure that the correct calibration file and analysis method is associated with each point. Once this has been confirmed, press the START icon to begin analysis.

Note: - New quartz and nickel boats are initially cleaned by batching them in vycor dishes and placing them inside an 850$^{o}$C muffle furnace for 10 minutes. The analysis process cleans the boats each time that they are used.. Residue, however, remains in the boats containing soil. For nickel boats transfer the loose residue to a small waste beaker. Any remaining can be wiped out with a thick pipe cleaner. The quartz has to

be washed with Reagent Grade Water and a Q-tip and rinsed well and dried with a kimwipe. Residue may remain but will not interfere with analysis.

12.6 Instrument Shut-Down

12.6.1 Hit the On/Off button.

12.6.2 Close the Oxygen toggle switch by the instrument.

12.6.3 Turn off the High purity oxygen gas tank.

## 13. Data Analysis and Calculations

NOTE: easyDOC3 Data System: - After samples are analyzed, the easyDOC3 Windows-based software will produce a report containing both raw data and calculated results in both ng and mg/kg of Mercury. The data is then converted into a macro where the % SOLA calculation for each sample is added to this spreadsheet.

13.1 Log in to computer

13.2 Click 2X on easyDOC3 icon

13.3 Click open (upper left screen)

13.4 Click My Computer (middle screen)

13.5 Click 2X Swissmemory (E:)

13.6 Click data and select desired file and click open

13.7 Click green X to transfer data to an excel spreadsheet. Save as y.xls (last file)

13.8 Minimize the 2 screens and click on Windows Explorer/x file/EPA DMA DATA/1ADMAMACRO)

13.8.1 Enable Macros

13.8.2 Click Tools

13.8.3 Click Macros

13.8.4 Click Macro

13.8.5 Click Run - Screen will prompt you for:
Date
Project Name
Project #
Project File Name (Project Name, Project #, Date)
Analyst

13.8.6 % Solids (in decimal form) must be entered for each soil sample.

13.8.7 If, the page break indicates that more than one page is needed for the data, to headline the top of each page.. Click on 1 and drag down to 10. Right click copy. Scroll down to first line after break 1 and click. Click Insert copied cells. This must be repeated for each additional page.

13.8.8 Access QA/QC Sheet form lower tab and fill out.

13.8.9 Save data to X file/EPA DMA DATA: e.g. DMA Raw and Calculated Data REMAP #08070008 100709.

13.8.10 Print Calculated Data and QA/QC Sheet.

## 14. Method Performance

An initial demonstration of capability (DOC) must be performed each time there is a significant change in the chemistry of the method, a major modification to an existing instrument, or a new instrument is installed. A DOC is performed by each analyst designated to analyze samples using this method. An annual check must subsequently be performed and documented for each analyst using this method.

14.1 Accuracy and Precision

14.1.1 Initial Demonstration of Capability

An initial demonstration of capability study must be conducted for this method for each analyst using this method. The study consists of the analysis of four standards which are from a source independent of the standard curve. The results of the standards must be within the acceptance criteria supplied by the manufacturer or within 20% if none are specified. The % RSD should be within 20%. The results of the accuracy and precision study (true value, % recovery, standard deviation and

% RSD) are maintained by the Quality Assurance Officer for each analyst and are located in the Central Branch File.

14.1.2 Continuing Demonstration of Capability

An annual continuing demonstration of capability study must be performed and documented. It may consist of either successfully analyzing a PT sample or analyzing 2 sets of AQC standards to within control limits as stated in section 14.1.1. The results of the continuing accuracy and precision study (true value, % recovery, standard deviation and % RSD or final report from the PT provider) are maintained by the Quality Assurance Officer for each analyst and are located in the Central Branch File.

14.2 Method Detection Limit (MDL)

An MDL Study must be conducted for this method. The study is based on the requirements listed in 40 CFR Part 136 Appendix B. Specific procedures for conducting an MDL study can be found in SOP # G-8. The MDL Study comprised the analysis of seven reagent grade water samples fortified at a level between 2-3x the detection limit. The results of the MDL determination (true value, average concentration, standard deviation and calculated MDL) are maintained by the Quality Assurance Officer for each method and are located in the Central Branch File.

14.3 Limit of Quantitation (LOQ)

The Laboratory performs a Limit of Quantitation (LOQ) study on an annual basis for analytes associated with chemistry methods, The validity of LOQ is confirmed by successful analysis of Laboratory Fortified Blank (LFB) at approximately 2X the reporting limit. The recovery of each is reviewed and approved by the Laboratory Management. A summary of all LOQ study performance is maintained in the Laboratory Central File.

## 15. Quality Control

If QC criteria provided in this method are not achieved, then corrective action(s) should be implemented. This may include sample re-analysis as determined by existing laboratory policy and/or in consult with lab management and QAO.

15.1 Calibration Curve

Primary Calibration:

Acceptance Criteria - A minimum of 5 standards and a blank for the lower curve followed by 4 standards for the higher curve are analyzed. The correlation coefficient of each curve must be ≥0.995. This calibration curve can now be used, repeatedly, until any of the QC checks (15.2 & 15.3), listed below, fail.

Corrective Action - If the correlation coefficient for either curve is <0.995, the calibration is disallowed. The calibration must be terminated, and repeated after correcting the problem.

15.2 Instrument Performance Check (IPC) or Initial Calibration Verification (ICV).

Acceptance Criteria - A low concentration (10ng Hg) ICV standard (to verify the low curve) and a high concentration (250ng Hg) ICV standard (to verify the high curve) are analyzed from a separate identifiable source (different vendor or different lot number from that used for calibration standards) at the beginning of each sample run, immediately following the initial 3 blank checks and the low check 1ng standard. The results of the IPC/ICV solutions must be within ±10% of their true value.

Corrective Action - If the results cannot be verified within the specified limits, re-analyze the IPC/ICV solution(s). If the results of the second analysis are not within the acceptance limits, the analysis must be discontinued and a new primary curve must be generated.

15.3 Low Check 1ng Standard

Acceptance Criteria - Analyze the Low Check 1ng calibration standard immediately following the blanks at the beginning of the run and after the CCB at the end of the run.. The % recovery of the Low Check 1 ng standard must be within ±30% of the true value.

Corrective Action - If the Low Check 1ng cannot be verified within the specified limits, reanalyze the solution. If the results of the second analysis are not within the acceptance limits, the analysis must be discontinued and a new primary curve must be generated.

**1**5.4 Instrument Performance Check (IPC) or Continuing Calibration Verification (CCV)

Acceptance Criteria - Analyze the IPC/CCV solution, from the same source as that used for the calibration standards, after every tenth sample (or more frequently, if required) and at the end of the sample run. The results of each IPC/CCV solution must be within ±20% of the true value.

Corrective Action - If the calibration cannot be verified within the specified limits, re-analyze the IPC/CCV solution. If the results of the second analysis of the IPC/CCV solution are not within the acceptance limits, the analysis must be discontinued, the cause determined and/or in the case of drift the instrument re-calibrated. All samples following the last acceptable IPC/CCV solution must be reanalyzed.

15.5 Initial Calibration Blank/Continuing Calibration Blank (ICB/CCB)/Blank Check

Acceptance Criteria - Analyze 3 Blank Checks (to assure that the system is clean), at the beginning of every run, after the ICV 250ng and at the end of the run. The third replicate of each group must be shown to be <1ng Hg. The third Blank of the second group will be named ICB. The CCB will precede every CCV All ICB/CCB results must be < the |Reporting Limit|. In addition, a minimum of three blanks must be analyzed at the beginning of the run and three at the end of the run in order to assure that the system is clean.

Corrective Action - If the result of the ICB/CCB is > |Reporting Limit|, the analysis should be stopped, the problem identified, and the ICB/CCB reanalyzed. If the ICB/CCB results remain > |Reporting Limit|, the instrument must be recalibrated.

NOTE: Preparatory Blank (PB) or Method Blank is the same as the ICB/CCB for this method and need not be run as a separate determination.

15.7 Laboratory Fortified Blank (LFB) or Aqueous Laboratory Control Samples (LCS-Aqueous) and Solid Laboratory Control Sample (LCS-Solid)

Acceptance Criteria - Analyze two LFB samples with each batch of aqueous samples of 20 or less. Calculate accuracy as percent recovery using the following equation:

$$\%\ Recovery = \frac{LFB}{s}\ X\ 100$$

where: LFB = laboratory fortified blank
s = concentration equivalent of mercury added to fortify the LRB solution.

Analyze two solid LCS samples with each batch of solid samples of 20 or less. Calculate accuracy as percent recovery using the following equation:

$$\%\ Recovery = \frac{Actual\ Value, mg/Kg}{True\ Value, mg/Kg}\ X\ 100$$

True Value for the solid LCS is available through the certificate of analysis supplied by the vendor. The % recovery of the aqueous LCS must be within ±20% of the true value. The % recovery of the solid LCS must be within ±25% of the true value or within the limits established by the vendor. The relative percent difference (RPD) of the duplicates should not exceed 20% for aqueous samples and 25% for solid samples.

Corrective Action - If the % recovery or %RPD results are outside the required control limits, the affected samples should be reprepared and reanalyzed. If the samples cannot be reprepared, then all affected sample results must be qualified accordingly.

15.8 Laboratory Fortified Matrix (LFM)/Matrix Spike(MS) Recovery

Acceptance Criteria - One Laboratory Fortified Matrix (LFM)/Matrix Spike (MS) is prepared for each matrix per project with at least one MS per batch of 20 or fewer samples. The LFM/MS aliquot must be a duplicate of the aliquot used for sample analysis. When possible, the concentration should be the same as that added to the aqueous LFB/LCS, but should not exceed the midpoint concentration of the calibration curve. Calculate the percent recovery, corrected for background concentration measured in the unfortified sample aliquot, and compare these values to the control limits to the designated matrices' recovery ranges : ±20% for aqueous samples; ±25% for solid samples (soils, sediment, and NAPL); and ±50% for sludge and biological tissue samples. Percent recovery is calculated using the following equation:

$$R = \frac{C_s - C}{s}\ X\ 100$$

where:
R = percent recovery,
Cs = fortified sample concentration,
C = sample background concentration, and
S = conc. equivalent of metal added to sample.

Corrective Action - If %Recovery of the MS is outside the required control limits, and the laboratory performance is shown to be in control, the recovery problem encountered is judged to be matrix related, not system related. The native sample result of the sample used to produce the MS must be qualified accordingly.

The % recovery of the MS is not evaluated if the result of the unfortified sample concentration is $\geq$1.0x the level used to fortify the sample.

## 16. Reporting and Validation

16.1 Reporting Limits

The reporting limits are calculated based on the concentration of the lowest calibration standard analyzed:, i.e. 0.01mg/kg. All reporting values should be rounded to 2 significant figures.

16.2 Sample Data Package

The sample data package should include but not limited to the following:

16.2.1 Mercury DMA QA/QC Checklist with all relevant information entered;

16.2.2 Copies of Log Book entries of DMA Instrument Analysis Log and % Solids

16.2.3 Raw Data

16.2.4 Calculated Data

16.2.5. Cross Reference Form

16.2.6 LCS Certificate of Analysis, if required

16.3 Laboratory Information Management System (LIMS)

The analyst enters the data on the LIMS under the appropriate analytical codes.

16.4 Data Validation

The data package is given to the reviewer. The review is done by a peer who was not involved in the analysis. Upon completion of the review, including validation of all the appropriate codes in the LIMS for the particular project(s), the data reviewer will sign

and date the QA/QC checklist.

16.5 Data Records

All project records associated with the data package are filed under one designated project number file. All other projects associated with the data package are referenced to this designated project number file via a "cross reference form". The cross reference form is placed in each of the project files that were associated with the data package.

The data package is placed in the bin identified for the designated project file. The records for this designated project file are filed in our locked record cabinets once all data from the project, e.g., non-metal inorganic data, organic data, microbiology data, etc. have been reviewed by the appropriate staff.

## 16. Pollution Prevention

16.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the USEPA recommends recycling as the next best option.

16.2 The quantity of chemicals purchased should be based on expected usage during it's shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

16.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult Less is Better: Laboratory Chemical and Management for Waste Reduction, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202)872-4477.

## 17. Waste Management

The USEPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process waste should be characterized and disposed of in an acceptable manner. The Agency urges

laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the Region 2 SOP G-6, "*Disposal of Samples and Hazardous Wastes".*

## 18. References


- U. S. Environmental Protection Agency. *"Mercury in Solids and Solutions by Thermal Decomposition Amalgamation and Atomic Absorption Spectrophotometry,"* Method 7473, Revision 0, January 1998.

- U. S. Environmental Protection Agency. Region 2, SOP C-110 *"Determination of Mercury in Aqueous, Soil/ Sediment, Sludge, Waste Oil/Organic Solvents, TCLP Extracts and Biological Tissue Matrices by Cold Vapor Atomic Absorption Spectrometry,"* Revision 2.1 March 2009.

- U. S. Environmental Protection Agency, Region 2, SOP G-6 "*Disposal of Samples and Hazardous Wastes."*

- U. S. Environmental Protection Agency, Region 2, SOP G-15 *"Laboratory Definitions and Data Qualifiers.*"

- U. S. Environmental Protection Agency, Region 2, SOP G-23 *"PERCENT DRY SOLIDS"*

- SW846 7471B

- U.S. Environmental Protection Agency, Region 2, SOP G-8 "Laboratory Policy for the Determination of Method Detection Limits (MDLs)."

- U. S. Environmental Protection Agency, Region 9, SOP 535 "Analysis of Mercury in Solids By Thermal Decomposition and AAS, Revision 2 April 2009.